



EVALUATION OF HIGH-TEMPERATURE LUBRICANTS FOR LOW-HEAT REJECTION DIESEL ENGINES

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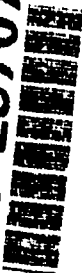
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19. ABSTRACT (Continue on reverse if necessary and identify by block number) A single-cylinder diesel engine was modified to simulate a low-heat rejection (LHR) engine, and it was used to develop lubrication requirements for future Army LHR diesel engines. Several high-temperature lubricant (HTL) candidates were evaluated, and the simulated LHR engine discriminated HTL deposition performance over a range of engine cylinder wall temperatures (CWTs). Three HTLs were identified that had promising performance at CWTs of 600°F (316°C), while none were adequate at 650°F (343°C). Oil was collected and analyzed from the ring zone of the simulated LHR engine. Oil degradation was as much as 3.7 times more severe in the ring zone as compared to the oil sump. Preliminary oxidation and friction-wear bench tests were investigated. New and used oil analyses flow charts were developed, and analytical techniques to separate and identify HTL additives and base stocks were developed.						
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EXECUTIVE SUMMARY

Problems and Objectives: Future engines for powering U.S. Army ground equipment are expected to require improved or even novel lubricants. Engine oil will be exposed to severe high-temperature environments. Current engine lubricant technology (MIL-L-2104F) is inadequate for future low-heat rejection (LHR) engine requirements such as the DDC 8V-71T LHR, Cummins AIPS engine, and others. The program objective was to develop lubrication requirements for future U.S. Army ground vehicle engine and transmission systems.

Importance of Project: A key limiting technology in the development of future LHR engines for the U.S. Army is the ability of the engine oil to function at elevated temperatures. Requirements for high engine oil temperature exceed the ability of current generation oils in the areas of thermal/oxidative stability and low-deposition rates.

Technical Approach: The approach is to develop requirements for high-temperature lubricants (HTL) that will encourage industry to develop improved HTLs. A single-cylinder simulated LHR engine was used to identify deficiencies in current oils, to develop HTL requirements, and to screen candidate HTLs. Concurrently, bench-scale screening requirements and HTL compositional analysis techniques were developed.

Accomplishments: A single-cylinder diesel engine was modified to simulate a low-heat rejection engine, and it was used to develop lubrication requirements for future U.S. Army LHR diesel engines. Several high-temperature lubricant candidates were evaluated, and the simulated LHR engine discriminated HTL deposition performance over a range of engine cylinder wall temperatures (CWTs). Three HTLs were identified that had promising performance at CWTs of 600°F (316°C), while none were adequate at 650°F (343°C). Oil was collected and analyzed from the ring zone of the simulated LHR engine. Oil degradation was as much as 3.7 times more severe in the ring zone as compared to the oil sump. Preliminary oxidation and friction-wear bench tests were investigated. New and used oil analyses flow charts were developed, and analytical techniques to separate and identify HTL additives and base stocks were developed.

Military Impact: Development of adequate high-temperature lubricants will allow all the benefits and payoffs of minimum-cooled diesel engines to be realized. The payoffs include improved specific fuel consumption, increased vehicle power density, reduced engine size, and reduced cooling maintenance requirements.

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I. INTRODUCTION

Low-heat rejection (LHR) diesel engines, also referred to as adiabatic engines, and other high-temperature, high-output diesel engines are being considered by the U.S. Army as power plants for future ground vehicles. As indicated in the Bibliography at the end of this report, many researchers have been involved in attempting to solve the technical challenges of developing adiabatic diesel engines. The insulated LHR turbocompounded engine is of particular interest to the Army. This engine configuration affords many important advantages of military interest. These advantages include compact engine size, lower engine weight/power output, less smoke, and improved specific fuel consumption.(1-13)* An LHR engine that can operate without a conventional liquid cooling system would also offer the Army the advantages of reduced combat vulnerability and decreased maintenance requirements.

By retaining thermal energy within the engine, LHR engines produce a greater thermal stress on the liquid lubricant. Top ring reversal (TRR) temperatures in an LHR engine can vary from 370°C to more than 560°C.(9-14) In previous work (7), BFLRF evaluated a wide range of petroleum and synthetic oils at elevated operating temperatures in a single-cylinder, simulated LHR engine. Of these oils, four are included in this program. The following engine oil deficiencies were observed when operating at 600° to 650°F (316° to 343°C) average cylinder wall temperature (CWT): excessive oil oxidation, which caused severe oil thickening and even oil solidification within 50 hours; corrosive attack of engine bearings; very high oil consumption; and unacceptable engine deposits that resulted in ring sticking.

The objective of this Army research program is to develop lubrication requirements for new Army ground vehicle engine systems such as the LHR diesel engine. The approach is to determine the deficiencies and limits of current generation commercially available, potential high-temperature lubricants (HTLs) in a simulated LHR diesel engine environment. These limits will lead to the definition of HTL performance requirements. Concurrently, bench-scale screening techniques are being developed to assist in the development of an optimized HTL. Only liquid

* Underscored numbers in parentheses refer to the list of references at the end of this report.

lubricants were considered during this project; however, other investigations conducted by BFLRF address the feasibility of using solid lubricants and other lubrication systems.(15,16)

II. SIMULATED LOW-HEAT REJECTION ENGINE

A. VM Model SU1051 Engine Description

In BFLRFs previous work, a highly modified single-cylinder CLR-D diesel engine was operated at high cylinder wall temperatures to simulate the temperature environment of an adiabatic diesel engine.(7) The CLR-D had rectangular top and second compression rings, which were conducive to ring sticking. Because many modern diesel engines use keystone compression rings to combat ring sticking, BFLRF required a small single-cylinder, four-cycle, direct-injection diesel engine with a keystone top compression ring. The VM Model SU1051 engine met the requirements and was obtained for the current program. Characteristics of the air-cooled VM SU1051 diesel engine are presented in TABLE 1.

TABLE 1. VM SU1051 Engine Characteristics

Displacement, cu in. (cm ³)	58 (952)
Bore, in. (mm)	4.133 (105)
Stroke, in. (mm)	4.330 (110)
Compression Ratio	17:1
Piston	Aluminum, 4-ring
Piston Rings	
Top Compression	Keystone, tapered, chrome
Second Compression	Square-faced, cast iron, inside chamfer
Third Compression	Cast iron
Oil Control/Scraper	Cast iron with expander
Cylinder Bore	Cast iron
Oil Capacity	3 qt (3.4 Liters)

B. Engine Modifications

Several modifications were made to the VM diesel engine to allow operation at increased cylinder wall temperatures. The modifications are summarized in TABLE 2. The first modification involved removing the cooling fan and then grinding the cooling fins from the cylinder bore area. Next, iron-constantan thermocouples were installed in the cylinder bore surface area to

**TABLE 2. VM Diesel Engine Modifications
for High-Temperature Operation**

- Cylinder Area Uncooled (Fins Removed)
- Thermocouples Measure Cylinder Surface Temperature
- Increased Piston/Cylinder Bore Clearance
- Al Sleeve With Electric Heaters Fitted Around Cylinder to Increase/Control Cylinder Temperature
- Increased Lubrication of Piston Pin
- Injector Area Cooled (Compressed Air)
- Engine Oil Cooled 132°C (270°F)

monitor the temperature of the area exposed to lubricating oil film. The thermocouples were installed by drilling through the cylinder wall and welding the constantan wire of the thermocouple flush to the internal cylinder surface.⁽¹⁷⁾ The locations of the ten cylinder bore thermocouples are shown in Fig. 1. The specific thermocouple locations are shown with respect to the piston at top and bottom dead center in Fig. 2.

The cylinder bore thermocouples were calibrated by placing the entire assembly in an oven and ramping the oven temperature up to 700°F (371°C). The cylinder bore thermocouples generally read within 10°F of the known oven temperature; thus, no correction factor was applied.

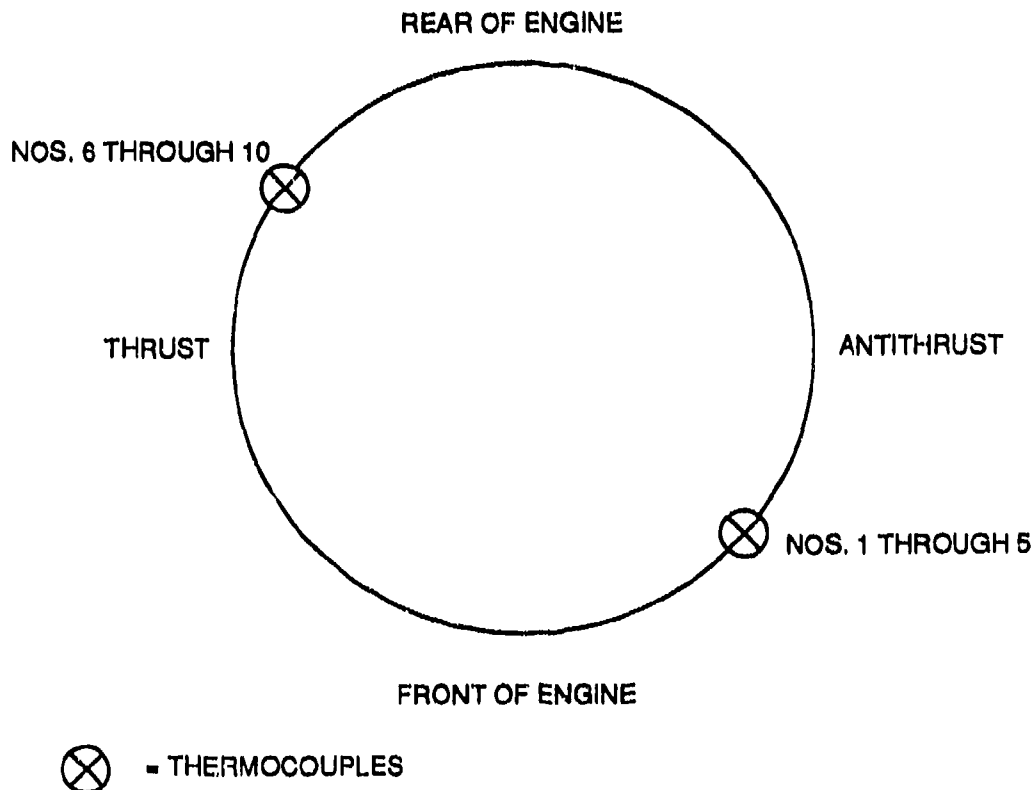


Figure 1. Thermocouple locations

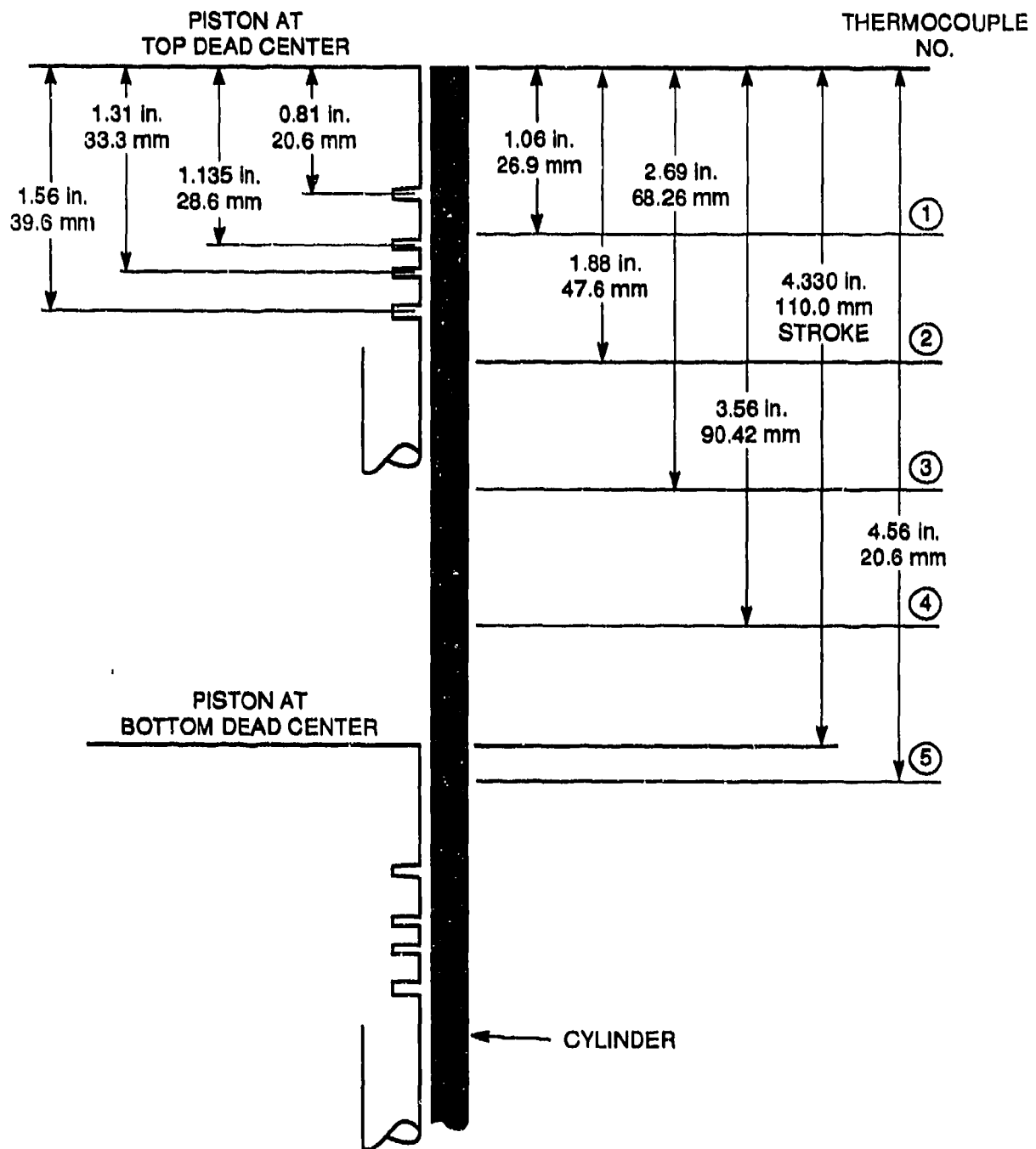


Figure 2. Specific thermocouple locations

As in BFLRFs previous work, an aluminum sleeve containing electric heating elements was fitted around the cylinder to increase and control cylinder temperature. A schematic of the aluminum heating sleeve is presented in Fig. 3. Five electric heating elements (750 watts each) were installed in the 0.75-in. (1.9 cm) thick aluminum sleeve.

Other modifications were made to increase the lubrication of the piston pin. The wrist pin was plugged at each end and grooved inward from each end with holes drilled in the grooved area and at the centerline of the pin as shown in Fig. 4.

The piston/cylinder clearance was modified to allow for increased thermal expansion by machining the piston as shown in Fig. 5 to obtain an average clearance of 0.0185 in. (0.47 mm). The cylinder bore was reused for several tests, while for most of the tests a new piston was used. As the cylinder bore increased from test to test, the piston/cylinder clearance was maintained approximately constant by varying the amount of material machined from the new piston.

Other modifications included using compressed shop air to cool the fuel injector area, which helped to prevent injector thermal fouling, and the use of an engine oil cooler. Engine sump oil temperature was maintained at 270°F (132°C) maximum. A photograph of the modified VM diesel engine is presented in Fig. 6.

C. Test Fuel

The base fuel was Reference No. 2 diesel fuel supplied by Howell Hydrocarbons, Inc. of San Antonio, TX. The specification requirements for this fuel, commonly referred to as "Cat fuel," are set forth in section 5.2, methods 354 and 355 of Federal Test Method Standard (FTMS) 791C and described in Appendix F of ASTM STP 509A, Part I and II.(18) This test fuel is a straight-run, mid-range natural sulfur fuel manufactured under closely controlled refinery operation to minimize batch-to-batch compositional and physical property deviations. Properties of the test fuel are given in TABLE 3.

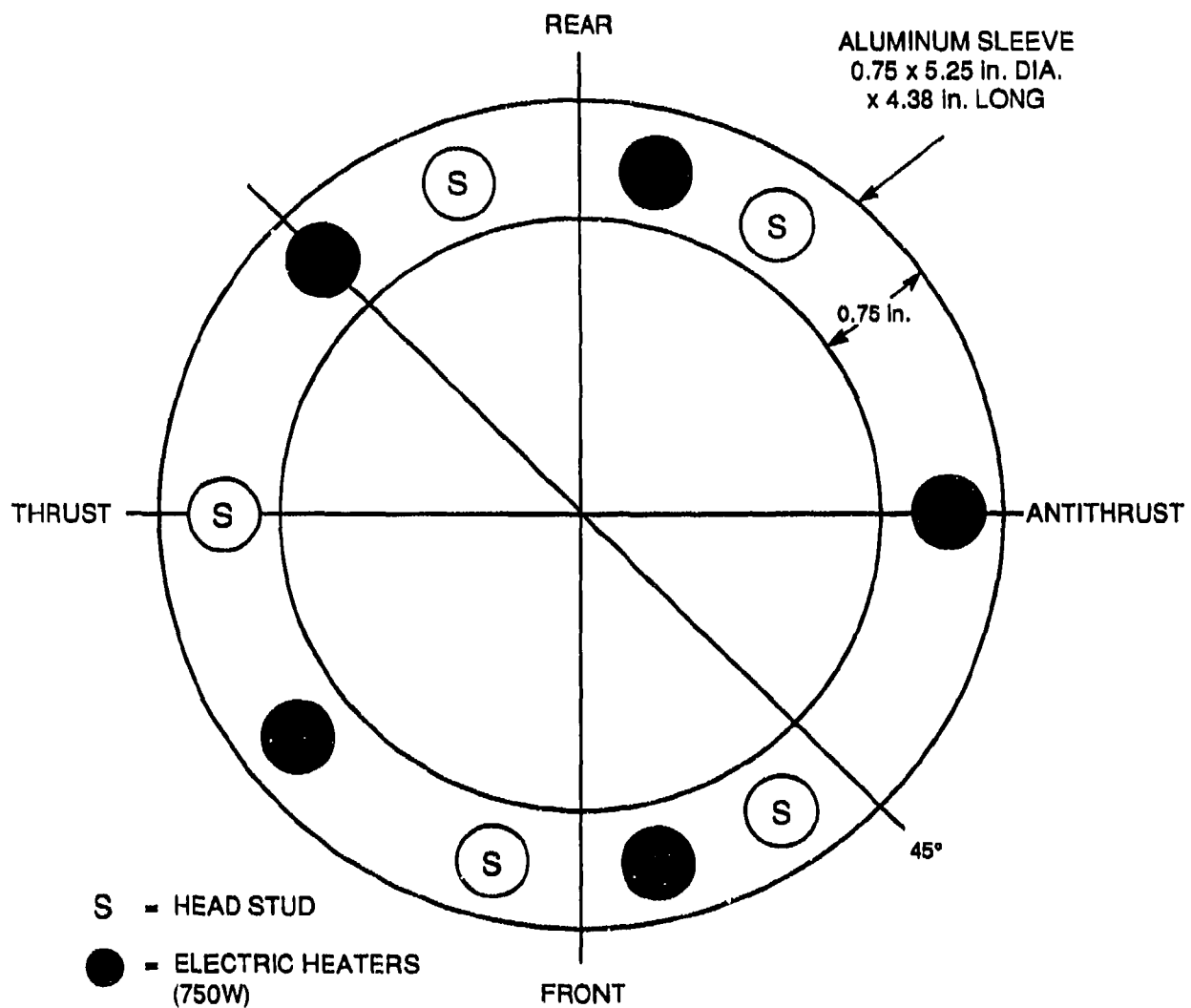


Figure 3. Aluminum heating sleeve

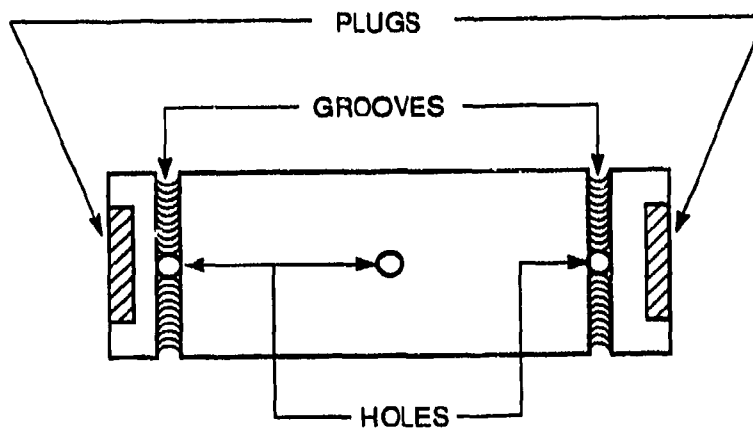
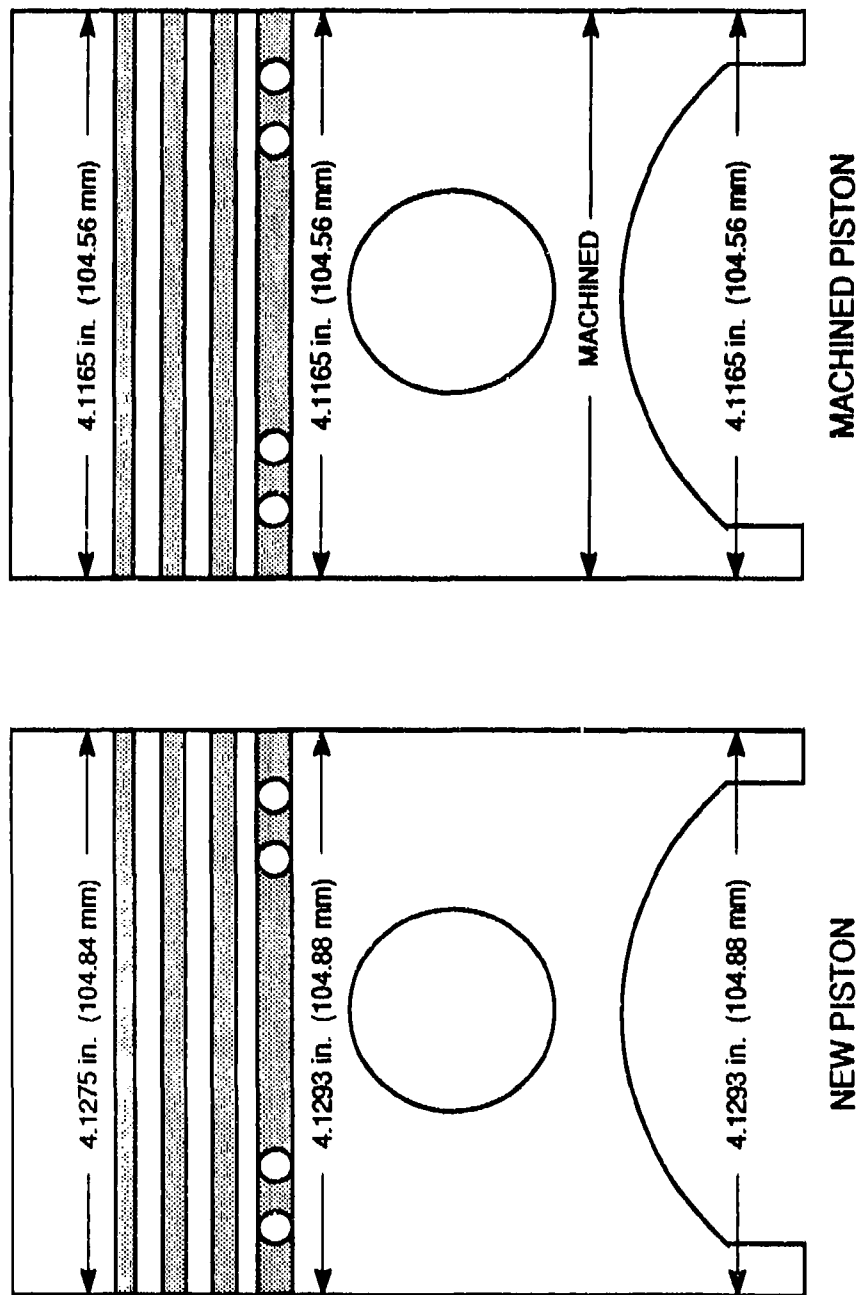
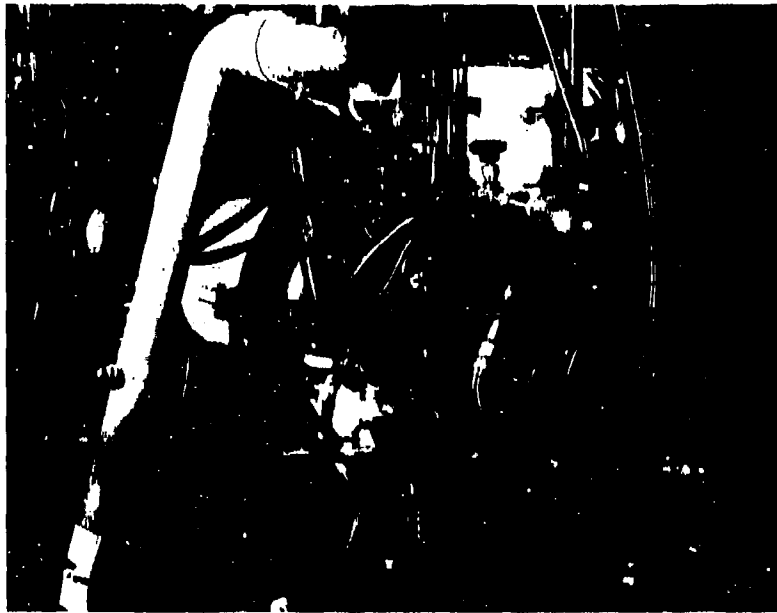


Figure 4. Wrist pin

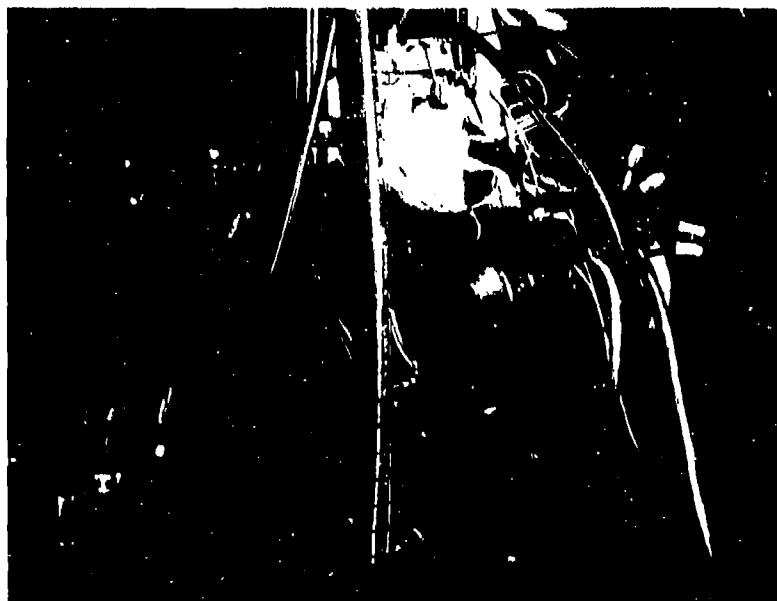


	NEW PISTON	MACHINED PISTON
CYLINDER BORE, in. (mm)	4.1350 (105.03)	4.1350 (105.03)
PISTON, in. (mm)	4.1293 (104.88)	4.1165 (104.56)
PISTON/CYLINDER CLEARANCE, in. (mm)	0.0057 (0.1448)	0.0185 (0.4699)

Figure 5. VM piston



a. Left



b. Right

Figure 6. Modified VM diesel engine

TABLE 3. Test Fuel Analysis

Properties	ASTM Method No.	Reference No. 2 DF	
		Test Fuel	Specification ^a
Gravity, API°	D 287	34.5	Record
Viscosity, cSt, 38°C (100°F)	D 445	3.3	1.6 to 4.5
Flash Point, °C (°F)	D 93	85 (185)	37.8 (100) min
Cloud Point, °C (°F)	D 2500	-2.0 (+28)	Record
Pour Point, °C (°F)	D 97	-12 (+10)	-6.7 (+20) max
Water and Sediment, vol%	D 1796	0.0	0.5 max
Carbon Residue, wt%	D 524	0.10	0.20 max
Sulfur, wt%	D 129	0.41	0.35 min
Acid No., mg KOH/g	D 664	0.0	Record
Aniline Point, °C (°F)	D 611	63 (145)	Record
Copper Corrosion	D 130	1A	No. 2 max
Distillation, °C (°F)	D 86		
Initial Boiling Point		207 (405)	Record
10%		241 (465)	Record
50%		273 (524)	260 (500) min
90%		317 (603)	316 to 338 (600 to 640)
End Point		348 (658)	343 to 366 (650 to 690)
Cetane No.	D 613	52	0 to 45
Net Heat of Combustion			
MJ/kg (Btu/lb)	D 240	42.13 (18,130)	Record
Ash, wt%	D 482	0.006	0.01 max

a = ASTM STP 509A, Part I and II, Appendix F.

D. Engine Operating Conditions

Typical engine operating conditions used for making HTL evaluations are shown in TABLE 4. Cylinder wall temperatures varied, depending on the amount of supplemental heat added to the cylinder bore area. With no supplemental heat added, the area reached a temperature of 470°F (243°C). The maximum average cylinder area temperature was 650°F (343°C) when the supplemental heat system was operated at maximum. The average cylinder wall temperature of 650°F (343°C) was obtained as shown in TABLE 5.

TABLE 4. Typical Operating Conditions of Uncooled VM Diesel Engine

Rpm	2000
Air/Fuel Ratio	25:1
Power, Obs Hp	9.5
BSFC, lb/Bhp • hr	0.421
Temperatures, °F (°C)	
Typical Cylinder Wall (Interior Surface)	635 (335)
Oil Gallery	270 (132) With Oil Cooler
Exhaust	975 (524)

TABLE 5. VM Engine Cylinder Surface Temperatures

	<u>Heated</u>
Engine, rpm	2000
Air/Fuel Ratio	25:1
Load, lb	27
Exhaust Temperature, °F (°C)	975 (524)
Thermocouple Number Temperature, °F (°C)	
1 (Top) and 6	687 (364)
2 and 7	664 (351)
3 and 8	656 (347)
4 and 9	630 (332)
5 (Bottom) and 10	614 (323)
Average	650 (343)

III. HIGH-TEMPERATURE LUBRICANT EVALUATIONS

A. Introduction

Several high-temperature lubricants were evaluated in the modified VM diesel engine, which simulated the environment of the low-heat rejection engine. The results are grouped by oil and are not necessarily presented in chronological test order. Some oils were evaluated at a variety of average cylinder wall temperatures (CWTs). Each new oil was analyzed, and physical

inspection properties were determined. The VM engine tests were scheduled for a minimum duration of 50 hours, and some were stopped prematurely because of oil degradation or engine problems such as high blowby or high wear metals. At the end-of-test (EOT), the used oil was analyzed, and the engine was disassembled and inspected.

B. Ring Zone Oil Sampling Technique

A technique for obtaining an oil sample from the ring zone of a diesel engine was introduced by Richard.(19) Additional work concerning the composition of diesel engine ring zone oil was conducted by Fox, et al.(20) Fox found that ring groove oil had extensive base oil evaporation, a much higher oxidation level than sump oil, and, for many analytical inspection properties, a steady-state condition developed.(20) An understanding of lubricant degradation in the ring zone will be useful in formulating improved high-temperature lubricants (HTLs). The diesel engine can be viewed as a two-stage chemical reactor with respect to engine oil. One chemical reactor is the piston ring zone area of the engine. The conditions of this reactor include (1) a relatively small quantity of oil present in a thin film that is exposed to high temperatures and pressures, (2) reactive combustion byproduct species, and (3) fresh metallic catalytic surfaces. The other chemical reactor is the engine oil sump. Contaminated, partially degraded oil from the ring zone is transported to the oil sump where the oil has a longer residence time for degradation reactions to occur.

During this program, the ring zone oil sampling technique was adapted to the high-temperature VM diesel engine test bed. A stainless steel sampling tube [1/8 in. OD (3.18 mm)] was installed opposite the second compression ring position at top dead center, which is 15/16 in. (23.81 mm) down the cylinder wall from the top. This location was selected because of space constraints. Ring zone oil sampling was conducted for approximately 75 percent of the HTL evaluations.

C. Discussion

Each HTL that was evaluated in the high-temperature VM diesel engine will be discussed with respect to oil composition including base stocks and additives and oil performance in the engine

test. A total of seven diesel engine-type lubricants were evaluated, along with six synthetic ester gas turbine jet engine oils, and one exotic polyphenylether material.

1. Oil A

Oil A is a commercially available synthetic polyolester-based diesel engine oil that has a Society of Automotive Engineers (SAE) viscosity grade of 15W-30. This oil has been evaluated in many HTL research programs and functions as an "unofficial" baseline reference oil. Oil A has an additive system that contains calcium, magnesium, zinc, and phosphorus, and has a sulfated ash content of 0.73 wt%. Inspection properties for Oil A are shown in TABLE 6. A summary of the VM engine tests, which used Oil A, is shown in TABLE 7. The five evaluations of Oil A had average cylinder temperatures from 470° to 652°F (243° to 329°C). TABLE 8 shows the average engine operating conditions for these tests, along with the summarized test results and used oil properties. Test A-1 was operated for 163 hours without supplemental heat in the cylinder liner area and had an average cylinder wall temperature of 470°F (243°C). Unfortunately, the piston from this test was cleaned before it could be rated; therefore, no piston deposit data are available. The used sump oil was moderately degraded with a 59 percent increase in viscosity at 100°C, Total Acid Number (TAN) increase of 2.2, and a Total Base Number (TBN) decrease of 2.4. Wear metals were low, and coagulated insolubles were still below 1 wt%. Comparison of the used sump oil with the oil collected from the ring zone (TABLE 9) revealed that the ring zone oil was more severely degraded than the sump oil, except for viscosity increase, which was similar. Ring zone oil had substantially increased TAN and reduced TBN along with increased coagulated insolubles. Additive element concentrations were enhanced in the sump and ring zone.

Test A-2 was conducted at 607°F (319°C) average CWT for a scheduled 50 hours. During Test A-2, high blowby from ring sticking occurred at 14, 19, 32, 40, and 49 hours. This condition was corrected by a slight cool-down period at each occurrence. At the end-of-test, the used oil had slight to moderate increases in viscosity (32 percent at 100°C) and TAN (+2.1), which indicated that oil oxidation was not severe. The piston was heavily deposited (WTD = 497), and

TABLE 6. Properties of Oil A

K. Vis, at 40°C, cSt	61.87
K. Vis, at 100°C, cSt	9.62
Viscosity Index	138
Pour Point, °C	-31
Flash Point, °C	263
Gravity, API°	18.5
Sulfated Ash, wt%	0.73
TAN	2.3
TBN (D 664)	7.4
Elements, wt%	
Ba	NIL
Ca	0.05
Mg	0.09
Zn	0.09
P	0.06
S	0.27
N	0.091

TABLE 7. Summary of VM Engine Tests Using Oil A

<u>Test No.</u>	<u>Average Cylinder Wall Temp., °F (°C)</u>	<u>Test Hours</u>
A-1	470 (243)	163
A-2	607 (319)	50
A-3	633 (334)	47
A-4	652 (344)	19.5
A-5	652 (344)	8
		(Modified Engine)

TABLE 8. VM Engine Tests: Oil A

Test No.	A-1	A-2	A-3	A-4	A-5
<u>Operating Conditions</u>					
Avg Cyl Wall Temp., °F (°C)	470 (243)	607 (319)	633 (334)	652 (344)	652 (344)
Min Avg CWT, °F (°C)	426 (219)	592 (311)	540 (282)	617 (325)	589 (309)
Max Avg CWT, °F (°C)	565 (296)	675 (357)	702 (372)	692 (367)	689 (365)
Oil Temp., °F (°C)	270 (132)	270 (132)	271 (133)	271 (133)	272 (133)
Speed, rpm	2000	2000	2000	2000	2000
Torque, ft-lb	28	26	25	27	27
Oil Cons, lb/hr	0.179	0.99	1.04	0.412	0.57
<u>Results</u>					
Test Hours	163	50	46	19.5	8
Ring Sticking					
Top	NA*	Free	Free	Free	Free
Second	NA	100% CS**	100% CS	100% Stuck	No Ring
Third	NA	100% CS	100% CS	Free	100% CS
Deposits					
Piston WTD	NA	497	441	302	417
Piston Skirt Demerits					
Thrust	NA	4.8	3.8	2.5	2.0
Antithrust	NA	1.5	2.2	1.8	1.25
Other Distress	—	—	High Blowby	High Blowby Cleaned 2nd Ring at 14 hr	No No. 2 CR
<u>Used Lubricant Properties</u>					
K. Vis, at 40°C, cSt	114.13	91.11	82.92	129.69	100.20
K. Vis, at 40°C, % Increase	86	42	34	110	62
K. Vis, at 100°C, cSt	15.29	12.73	11.86	16.22	13.39
K. Vis, at 100°C, % Increase	59	32	23	69	39
TAN	4.5	4.4	3.0	4.6	4.5
TAN Change	+2.2	+2.1	+0.7	+2.3	+2.2
TBN	5.0	5.2	5.4	6.5	5.9
TBN Change	-2.4	-2.2	-2.0	-0.9	-1.5
Wear Metals, ppm					
Fe	59	15	92	40	42
Cu	<10	<10	<10	<10	<10
Pb	<60	<60	<60	<60	<60
Insolubles, wt%					
Pentane, B	0.90	0.12	0.07	0.15	0.20
Toluene, B	0.78	0.11	0.07	0.13	0.15

* NA = Not Available, piston cleaned before rated.

** CS = Cold Stuck.

TABLE 9. Analysis of Ring Zone Oil — Tests A-1 and A-3

Properties	New	Test A-1		Test A-3	
		163 Hr at 470°F (243°C) CWT		47 Hr at 630°F (332°C) CWT	
		Sump	Ring Zone	Sump	Ring Zone
K. Vis, at 100°C, cSt	9.62	15.3	15.9	11.86	26.6
TAN	2.3	4.5	7.7	3	17.6
TBN	7.4	5.0	1.9	5.4	0.1
Toluene Insolubles (Congulated)	NIL	0.8	1.5	0.7	IS*
Ca, wt%	0.05	0.10	0.09	0.08	0.10
Zn, wt%	0.09	0.17	0.17	0.14	0.18
Fe, ppm	NIL	59	44	92	92

* IS = Insufficient Samples.

the second and third rings were 100 percent cold stuck. The increase in average cylinder wall temperature from 470°F to 607°F (243° to 319°C) produced a 5.5-fold increase in oil consumption.

Test A-3 was conducted at an average CWT of 633°F (334°C). As with Test A-2, occasional high blowby was a problem encountered during this test, which was stopped at 47 hours. Condition of the used sump oil was similar to Test A-2. As shown in TABLE 9, the ring zone oil (RZO) from Test A-3 was substantially more degraded than the sump oil. The RZO had a TAN of 17.6 with virtually no reserve alkalinity left. Viscosity increase was approximately twice that of the sump oil. This test demonstrated the severe degradation of the oil that occurs in the ring zone of a simulated low-heat rejection diesel engine. Improved high-temperature diesel additive compositions are needed.

Test A-4 was conducted at an average CWT of 652°F (344°C). This test was stopped at 14 hours due to excessive blowby. When the engine was disassembled, the keystone-type top

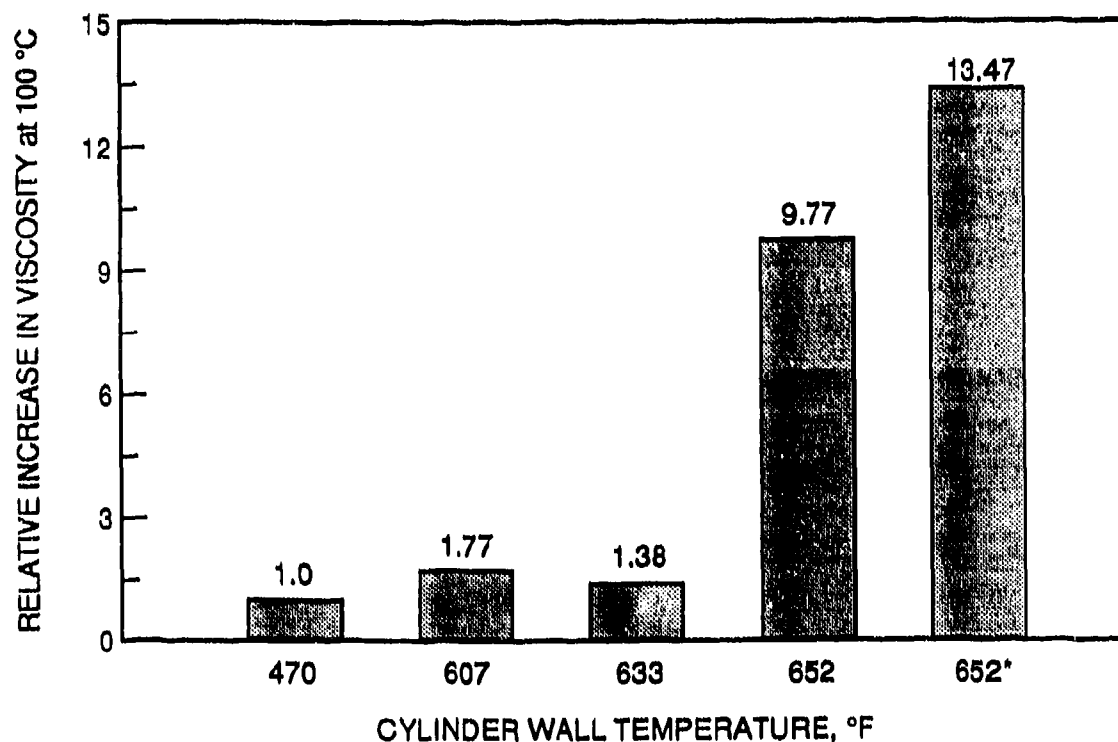
compression ring was free; however, it is believed that the sudden blowby increase may have been caused by partial sticking of this ring. In addition, the second compression ring was 100 percent stuck. The second ring was cleaned and replaced, and the engine test was continued. At 19.5 hours, a sudden increase in engine blowby occurred again, and the test was terminated. Inspection showed that the second ring was 100 percent stuck and possibly the top ring was sticking when hot. Used oil viscosity (100°C) had increased 69 percent; however, TAN had increased by only 2.3 and substantial reserve alkalinity remained. The high rate of oil consumption (0.4 lb/hr) and resulting makeup oil additions impacted the condition of the used oil at the end-of-test. While the oil consumption rate was not as high as Tests A-2 and A-3, it is postulated that the oil consumption rate of Test A-4 would increase with longer operating time.

Test A-5 was run without the nonkeystone-type second compression ring installed to determine if the sudden loss of blowby control was caused by the keystone-type top compression ring sticking. At 8 hours, loss in blowby control occurred, indicating that the top keystone ring was sticking when hot. As with Test A-4, viscosity increase was severe, especially considering the short duration of the test.

Test-to-test comparisons are difficult because the test durations vary. To aid in comparing CWT effects, a relative viscosity increase index was calculated as follows: the percent viscosity increase (100°C) was divided by test hours and then normalized against the lowest value [Test A-1, 470°F (243°C) CWT]. The results are plotted in a bar graph (Fig. 7), which shows that oil consumption increases dramatically above 633°F (334°C). Overall, Oil A was not suitable for use at CWTs above 600°F (316°C) because of excessive piston deposits, which impacted engine operation by resulting in ring sticking and loss of engine blowby control.

2. OIL B

Oil B is a commercially available petroleum-based engine oil (SAE grade 20W-50), which is designed to meet the increased temperature demands of turbocharged gasoline engines. Oil B meets the requirements of API service classifications SF/CD. The additive chemistry of Oil B



* - Engine modified - No. 2 compression ring omitted

Figure 7. Relative increase, K. Vls at 100°C, with Oil A

consisted of calcium, magnesium, sodium detergent-dispersant components, zinc, and phosphorus. Five different batches of Oil B were obtained during this program. The analyses of each batch and a calculated average for each property are shown in TABLE 10. Average sulfated ash for Oil B was 1.07 wt%. A summary for the VM engine tests, which used Oil B, is presented in TABLE 11. The nine evaluations of Oil B were conducted at seven different cylinder wall temperatures, which varied from 470° to 648°F (243° to 342°C). TABLE 12 presents the average engine operating conditions for these tests, along with the summarized test results, and used oil properties.

Test B-1 was operated without supplemental heat in the cylinder liner area, and completed 89 hours at an average CWT of 470°F (243°C). The primary objective of Test B-1 was to determine the feasibility of collecting ring zone oil (RZO). This experiment was successful, and the feasibility of collecting RZO was demonstrated. Sump oil was changed at 56 hours when

TABLE 10. Properties of Oil B

Batch Properties	AL-13197-L	AL-13369-L	AL-13872-L	AL-14386-L	AL-14486-L	Average
K. Vis, cSt, at 40°C	191.38	183.88	186.21	180.54	201.98	188.90
100°C	20.97	20.09	20.23	19.95	21.06	20.46
Viscosity Index	130	127	126	128	124	127
Sulfated Ash, wt%	1.12	1.19	0.86	0.98	1.22	1.07
TAN	2.0	1.9	1.6	1.3	2.1	1.8
TBN	6.6	4.6	5.4	5.8	5.7	5.6
Elements, wt%						
Ba	NIL	NIL	NIL	NIL	NIL	NIL
Ca	0.10	0.11	0.10	0.09	0.12	0.10
Mg	0.05	0.05	0.04	0.04	0.04	0.04
Na	0.07	0.08	0.03	0.06	0.09	0.07
Zn	0.14	0.12	0.13	0.13	0.12	0.13
P	0.12	0.11	0.10	0.13	0.12	0.12
S	0.54	ND*	0.60	0.86	0.59	0.65
N	0.051	ND	0.048	ND	0.079	0.059
GCBPD, °C at wt% off						
1	329	ND	318	330	307	321
5	369	ND	371	382	368	373
10	388	ND	394	405	397	396
20	417	ND	425	430	428	425
30	443	ND	449	447	447	447
40	467	ND	468	463	464	466
50	491	ND	487	480	479	484
60	533	ND	507	500	496	509
70	>600	ND	535	526	521	ND
80	>600	ND	>600	>600	586	ND
90	>600	ND	>600	>600	>600	>600
95	>600	ND	<600	>600	>600	>600
Residue, wt%, 600°C	37.9	ND	23.3	20.1	19.4	25.2

* ND = Not Determined.

TABLE 11. Summary of VM Engine Tests Using Oil B

Test No.	Average Cylinder Wall Temp., °F (°C)	Test Hours
B-1	470 (243)	89
B-2	510 (266)	88
B-3A	550 (288)	39
B-3B	550 (288)	75
B-3C	550 (288)	105
B-4	593 (312)	96
B-5	608 (320)	102
B-6	636 (336)	41
B-7	648 (342)	27

TABLE 12. Summarized Test Results: Oil B

Test No.	B-1	B-2	B-3A	B-3B	B-3C	B-4	B-5	B-6	B-7
Operating Conditions									
Avg Cyl Wall Temp., °F (°C)	470 (243)	510 (266)	550 (288)	550 (288)	550 (288)	593 (312)	608 (320)	636 (336)	648 (342)
Min Avg CWT, °F (°C)	469 (243)	487 (253)	525 (274)	526 (274)	521 (272)	538 (281)	562 (294)	540 (282)	599 (315)
Max Avg CWT, °F (°C)	475 (246)	589 (309)	609 (321)	606 (319)	607 (319)	660 (349)	683 (362)	679 (359)	678 (359)
Oil Temp., °F (°C)	271 (133)	270 (132)	271 (133)	270 (132)	273 (134)	270 (132)	273 (134)	272 (133)	271 (133)
Speed, rpm	2000	2002	2000	2004	2043	2011	2006	2000	2000
Torque, ft-lb	28	27	26	27	25	24	24	26	23
Oil Cons, lb/hr	0.376	0.425	0.506	0.386	0.367	0.404	0.945	0.89	0.43
Results									
Test Hours	89	88	39	75	105	91	102	41	27
Compress Ring Sticking									
Top	Free	Free	Free	Free	ND*	Free	Free	Free	ND
Second	100% CS**	100% CS	100% CS	100% CS	ND	100% CS	100% CS	100% CS	ND
Third	Free	Free	15% CS	Free	ND	100% CS	75% CS	100% CS	ND
Deposits									
Piston WTD	214	324	273	440	ND	386	401	424	ND
Piston Skirt Deterits									
Thrust	0.5	2.9	1.8	2.8	ND	3.7	5.6	5.8	ND
Antihrust	0.2	2.0	1.5	2.0	ND	2.7	2.5	2.8	ND
Other Distress			High Blowby			OC at 45 hr	High Oil Cons	High Blowby	Rings Stuck at 15 hr
Used Lubricant Properties									
K. Vis, at 40°C, cSt	ND	1190.91	689.92	680.71	985.74	ND	414.09	378.17	478.6
K. Vis, at 40°C, % Increase	ND	531	271	278	388	ND	105	98	157
K. Vis, at 100°C, cSt	26.79†	69.99	48.66	47.84	62.57	84.63	34.95	31.27	36.16
K. Vis, at 100°C, % Increase	31	242	141	140	197	302	66	49	79
TAN	ND	4.2	4	4.6	3	ND	1.8	3.7	4.3
TAN Change	ND	2.4	2.4	3.3	0.9	ND	-0.3	1.7	2.7
TBN	ND	7.2	5.2	5.5	0.2	ND	4.9	5.3	4.9
TBN Change	ND	1.6	-0.2	-0.3	-5.5	ND	-0.8	-1.3	-0.5
Wear Metals, ppm									
Fe	25†	69	100	52	40	282	87	62	77
Cu	<10	<10	<10	<10	<10	<10	13	<10	<10
Pb	<60	<60	<60	<60	<60	<60	<60	<60	<60
Insolubles, wt%									
Pentane, B	ND	0.36	0.26	0.32	0.39	ND	0.13	0.32	0.31
Toluene, B	ND	0.34	0.24	0.26	0.37	ND	0.12	0.29	0.29

* ND = Not Determined.

** CS = Cold Stuck.

† = at 65 hours.

kinematic viscosity (K. Vis) at 100°C reached 32 cSt. At 89 hours, engine operation became erratic, and the test was terminated. The collected RZO had the following properties:

K. Vis, cSt, at	
100°C	42.97
40°C	571.44
Viscosity Index	122
TAN	3.1
TBN	4.0
Insolubles, coagulated, wt%	
Pentane	0.54
Toluene	0.48
Fe, ppm	43
Ca, wt%	0.16
Na, wt%	0.15
Zn, wt%	0.16

As shown above, the RZO had thickened considerably, and contained increased additive element concentrations. Because of the intermediate sump oil change, the end-of-test sump oil drain was not analyzed.

Test B-2 was conducted at an average CWT of 510°F (266°C), and completed 88 hours before being stopped because of oil viscosity increase (242 percent at 100°C). Engine disassembly revealed that the second compression ring was 100 percent cold stuck. Ring zone oil was collected during the first 72 hours of the test, and the RZO analyses are presented in TABLE 13, along with analyses of the sump oil. The Gas Chromatography Boiling Point Distributions (GCBPDs) show the removal of light ends of the base stock in both the sump oil and collected RZO. Oil residue, defined as material boiling greater than 600°C, increased from 25 percent in the new oil to 49 percent in the sump oil and 62 percent in the RZO. Viscosity increase at 100°C of the RZO was 309 percent. Additive content was concentrated in both the sump and RZO, which resulted in increased reserve alkalinity (TBN) in both used oils. Overall, oil degradation was slightly more severe for the RZO as compared to the sump oil.

TABLE 13. Ring Zone Oil Tests: Oil B

Test No. Hours CWT, °F (°C) Oil Properties	B-2		B-3B			B-5		B-7	
	88	0 to 72	75	0 to 26	26 to 75	102	0 to 102	27	0 to 27
	510 (266)		550 (288)			608 (320)		648 (342)	
	Sump	RZ	Sump	RZ	RZ	Sump	RZ	Sump	RZ
K. Vis, at 100°C, cSt	69.99	83.60	47.84	72.94	122.14	34.95	53.09	36.16	56.01
K. Vis, at 100°C, % Increase	242	309	134	256	497	66	159	79	174
TAN	4.2	4.5	4.6	4.4	6.6	1.8	3.9	4.3	10.0
TAN Change	+2.4	+2.7	+2.8	ND*	ND	-0.3	+2.1	+2.7	+8.2
TBN	7.2	7.4	5.5	6.5	6.8	4.9	5.6	4.9	0.6
TBN Change	+1.6	+1.8	-0.1	+0.9	+1.2	-0.8	0	-0.5	-5.0
DIR									
Ox Ab/Cm	152	108	96	68	116	38	234	75	113
Nitr Ab/Cm	32	8	16	8	4	4	12	0	4
GCBPD, °C at wt% off									
1	362	380	349	365	362	338	362	ND	ND
5	433	448	408	433	443	392	420	ND	ND
10	457	472	433	458	473	420	444	ND	ND
20	480	501	462	490	508	448	471	ND	ND
30	498	533	486	516	537	466	492	ND	ND
40	518	>600	509	549	600	483	515	ND	ND
50	569	>600	537	>600	>600	507	547	ND	ND
60	>600	>600	583	>600	>600	527	>600	ND	ND
70	>600	>600	>600	>600	>600	578	>600	ND	ND
80	>600	>600	>600	>600	>600	>600	>600	ND	ND
90	>600	>600	>600	>600	>600	>600	>600	ND	ND
Residue, wt%, 600°C	49.3	61.9	38.6	56.3	60.0	28.9	42.2	ND	ND
Elements, wt%									
Ca	0.20	0.30	0.23	0.31	0.33	0.24	0.36	0.20	0.16
Zn	0.27	0.29	0.22	0.23	0.28	0.21	0.31	0.23	0.17
N	0.124	0.172	0.076	0.141	0.166	0.104	0.131	0.068	0.059
Element, ppm									
Fe	69	83	52	68	71	87	104	77	425

* ND = Not Determined.

Oil B was evaluated at 550°F (288°C) CWT in three separate tests (B-3A, B-3B, B-3C) during the course of the program. Operation at these conditions served as a baseline check point. The three tests at 550°F (288°C) CWT lasted 39, 75, and 105 hours. The 39-hour test was stopped because of ring sticking and resultant high blowby and oil viscosity increase. Tests lasting 75 and 105 hours were stopped because of viscosity increase. Ring zone oil was collected and analyzed for Test B-3B as shown in TABLE 13 for periods of 0 to 26 and 26 to 75 hours. Compared to the sump oil, the 26- to 75-hour RZO sample had 3.7 times the viscosity increase, 1.6 times the GCBPD residue, and 1.2 times the Differential Infrared (DIR) oxidation level. These readings indicate that, in addition to oil oxidation, volatility loss of the oil was a major factor in the viscosity increase. Used oil iron content in the RZO at end-of-test was 1.4 times

the sump oil iron content. Overall at 550°F (288°C) CWT, RZO degradation was 1.2 to 3.7 times as severe as the sump oil degradation.

Tests B-4 and B-5 were conducted at 593°F (312°C) CWT for 91 hours and 608°F (320°C) CWT for 102 hours, respectively. Test B-4 had an oil change at 45 hours because of oil thickening, while Test B-5 was allowed to continue without an oil change because fresh oil was continually being added due to high oil consumption. Moderate to heavy piston deposits and severe oil thickening were observed for both tests. High used oil iron was observed during Test B-4 from ring and liner wear. RZO was collected and analyzed for Test B-5 (TABLE 13). RZO viscosity increase was 2.4 times that of the sump oil, and RZO oil oxidation by DIR was 6.2 times the sump oil. At 608°F (320°C) CWT, the RZO viscosity increase tended to be more oxidation related than oil evaporation, as the RZO oil had only 1.5 times the GCBPD residue of the sump oil.

Early in the program, Oil B was evaluated at 648°F (342°C) CWT (Test B-7). This test lasted only 27 hours and was terminated because of oil thickening and stuck rings. Overall, Oil B did not have adequate high-temperature performance for operation at increased CWTs [$>470^{\circ}\text{F}$ (243°C)].

3. Oil C and Oil CM

Oil C and a later modified version designated Oil CM are commercially available oils, which contain polyolester and polyalphaolefin base stocks. Properties of these two oils are presented in TABLE 14. Oil C is marketed as an SAE 5W-30 oil, while Oil CM is marketed as an SAE 5W-40 oil. Exact low-temperature viscosity grades were not determined. Oil C had a calcium-based additive system (6.3 TBN), while Oil CM contained a calcium/magnesium additive system (14 TBN) and had a higher sulfated ash content.

A summary of the VM engine tests, which used Oils C and CM, is presented in TABLE 15. Oil C was evaluated at 614°, 633°, and 630°F (323°, 334°, and 332°C) CWT. Summarized test

TABLE 14. Properties of Oil C and Oil CM

Oil ID	Oil C	Oil CM
Properties		
K. Vis, cSt, at 40°C	56.0	85.95
100°C	10.0	15.21
Viscosity Index	167	188
Flash Point, °C	224	241
TAN	3.0	3.5
TBN (D 664)	6.3	14.0
Sulfated Ash, wt%	1.10	1.45
Elements, wt%		
Ba	NIL	NIL
Ca	0.25	0.17
Mg	NIL	0.15
Na	ND	NIL
Zn	0.13	0.11
P	0.13	0.10
S	0.33	0.20
N	0.14	0.072
Evaporation at 191°C, wt% loss	7	13
GCBPD, °C at wt% off		
1	335	356
5	389	408
10	406	413
20	418	421
30	431	434
40	436	459
50	451	467
60	465	471
70	472	479
80	481	489
90	508	516
Residue, wt%, 600°C	0.4	1.3

TABLE 15. Summary of VM Engine Tests Using Oils C and CM

Test No.	Average Cylinder Wall Temp., °F (°C)	Test Hours
Oil C		
C-1	614 (323)	50
C-2	633 (334)	49
C-3	630 (332)	215
		(With Four Oil Changes)
Oil CM		
CM-1	550 (288)	69
CM-2	550 (288)	63
CM-3	625 (329)	53

results for Oil C are presented in TABLE 16. Test C-1 completed a scheduled 50 hours at 614°F (323°C) CWT with only an 18 percent increase in viscosity (K. Vis at 100°C) and a TAN increase of 6.4. Test C-2 [633°F (334°C)] CWT completed 49 test hours. The 20°F increase in CWT of Test C-2 caused a substantial increase in oil degradation compared to Test C-1. While K. Vis at

100°C increased by only 24 percent, TAN increased by 23.5, indicating that the synthetic ester oil was hydrolyzing. Test C-2 piston deposits were heavier at 548 WTD versus 392 WTD for Test C-1, and, in both tests, the second and third compression rings were cold stuck.

Test C-3 was run 215 hours to determine the effects of long-term operation at high CWT [624°F (329°C)] with oil changes when necessary. Oil changes were conducted at 84, 127, 157, and 197 hours. The piston was rated and cleaned at 84 and 157 hours and end-of-test. The WTD piston deposit ratings versus piston hours of operation at 625°F (329°C) CWT are shown in a bar graph in Fig. 8 and reveal that deposition accelerated rapidly after 73 hours of operation. Ring zone

TABLE 16. Summarized Test Results: Oil C

Test No.	C-1	C-2	C-3					
Operating Conditions								
Avg Cyl Wall Temp., °F (°C)	614 (323)	633 (334)	624 (329)					
Min Avg CWT, °F (°C)	535 (279)	547 (286)	560 (293)					
Max Avg CWT, °F (°C)	650 (343)	674 (357)	690 (366)					
Gallery Oil Temp., °F (°C)	269 (132)	271 (133)	272 (133)					
Speed, rpm	2000	2000	1997					
Torque, ft-lb	23	26	24					
Oil Consumption, lb/hr	0.54	0.72	0.68					
Results								
Test Hours	50	49	215*					
			Piston Hr					
			84	73	58			
Compression Ring Sticking								
Top	Free	Free	Free	Free	Free			
Second	100% CS**	100% CS	100% CS	100% CS	100% CS			
Third	100% CS	100% CS	100% CS	100% CS	100% CS			
Deposits								
Piston WTD	392	548	454	338	330			
Piston Skirt Demerits								
Thrust	3.7	5.8	4.8	5.3	5.6			
Antithrust	1.5	2.8	2.8	2.8	2.8			
Other Distress		Sluggish Oil Control Ring (OCR)	OCR 80% CS	OCR Sluggish	OCR 60% CS			
			Sump Oil, Oil Hr/Test Hr					Ring Zone
			24/108	43/127	30/157	40/197	18/215	Oil at 215 Hr
Used Lubricant Properties								
K. Vis, at 40°C, cSt	75.61	78.19	ND†	ND	ND	ND	ND	75.31
K. Vis, at 40°C, % Increase	35	40	ND	ND	ND	ND	ND	34
K. Vis, at 100°C, cSt	11.78	12.45	29.30	86.09	135.63	65.04	21.74	11.39
K. Vis, at 100°C, % Increase	18	24	193	760	1256	550	117	14
TAN	6.4	26.5	ND	ND	ND	ND	ND	6.8
TAN Change	+3.4	+23.5	ND	ND	ND	ND	ND	ND
TBN	4	3.5	ND	ND	ND	ND	ND	ND
TBN Change	-2.3	-2.8	ND	ND	ND	ND	ND	1.7
Wear Metals, ppm								
Fe	41	43	ND	ND	ND	ND	ND	68
Cu	<10	<10	ND	ND	ND	ND	ND	<10
Pb	<60	<60	ND	ND	ND	ND	ND	<60
Insolubles, wt%								
Pentane, B	0.13	0.20	ND	ND	ND	ND	ND	ND
Toluene, B	0.13	0.19	ND	ND	ND	ND	ND	ND

* Oil changes at 84, 127, 157, and 197 hr.

** CS = Cold Stuck.

† ND = Not Determined.

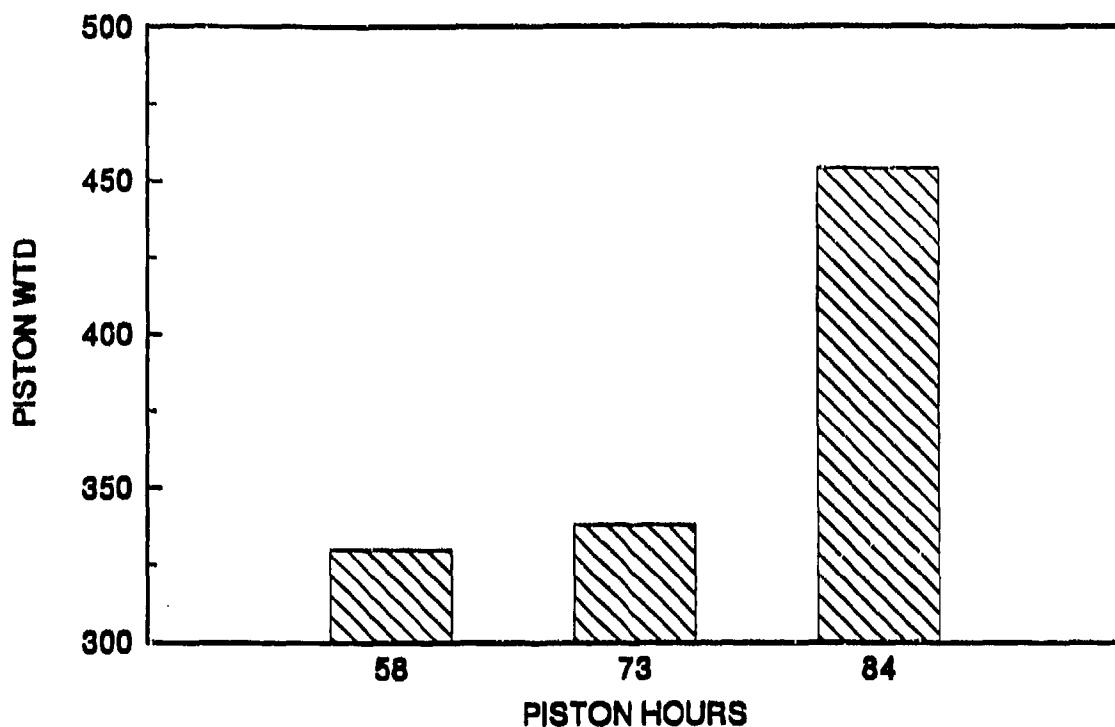


Figure 8. Piston WTD versus piston hours (Test C-3)

oil was collected; however, the collection tube apparently was plugged for most of the test because of the small quantity collected and the relatively unstressed properties of the RZO. Overall, Oil C had inadequate deposition characteristics at CWTs of $>600^{\circ}\text{F}$ (316°C), and adequate viscosity increase control for up to 50 hours at 614°F (323°C) CWT.

Oil CM was evaluated twice at 550°F (288°C) CWT and once at 625°F (329°C) CWT, with the summarized test results presented in TABLE 17, and the ring zone oil analyses in TABLE 18. A plot of oil viscosity increase with test hours is shown in Fig. 9. At 550°F CWT, Oil CM experienced very excessive viscosity increase in 63 hours and extremely excessive viscosity increase in 69 hours. TBN was concentrated in the used oil with Test CM-1 (69 hours) having a TBN of 38, and CM-2 having a TBN of 26.5. TAN increased to 12.3 during Test CM-1, indicating that the oil was degrading severely.

TABLE 17. Summarized Test Results: Oil CM

Test No.	<u>CM-1</u>	<u>CM-2</u>	<u>CM-3</u>
<u>Operating Conditions</u>			
Avg Cylinder Wall Temperature, °F (°C)	550 (288)	550 (288)	625 (329)
Min Avg Cyl Wall Temperature, °F (°C)	499 (259)	509 (265)	587 (308)
Max Avg Cyl Wall Temperature, °F (°C)	639 (337)	630 (332)	748 (398)
Gallery Oil Temperature, °F (°C)	269 (132)	270 (132)	270 (132)
Speed, rpm	2023	2035	2021
Torque, ft-lb	25	26	23
Oil Cons, lb/hr	0.47	0.37	0.71
<u>Results</u>			
Test Hours	69	63	53
Compress Ring Sticking			
Top	Free	Free	Free
Second	100% CS*	30% CS	100% CS
Third	Sluggish	Free	100% CS
Deposits			
Piston WTD	290	236	298
Piston Skirt Demerits			
Thrust	3.1	3.0	4.7
Antithrust	3.3	2.8	4.0
<u>Used Lubricant Properties</u>			
K. Vis, at 40°C, cSt	6034	753.67	2619.3
K. Vis, at 40°C, % Increase	7120	777	2816
K. Vis, at 100°C, cSt	295.2	121.9	143.7
K. Vis, at 100°C, % Increase	1840	701	845
TAN	12.3	7.8	12.3
TAN Change	+8.8	+4.3	+8.8
TBN	38.0	26.5	31.1
TBN Change	+24	+12.5	+17.1
Wear Metals, ppm			
Fe	104	57	251
Cu	8	4	11
Pb	15	6	16
Insolubles, wt%			
Pentane, B	0.45	0.18	1.38
Toluene, B	0.43	0.16	1.30
TGA Soot, wt%	2.9	1.0	ND**
Ca, wt%	0.64	0.53	1.02

* CS = Cold Stuck.

** ND = Not Determined.

TABLE 18. Ring Zone Oil Analyses: Oil CM

Test No. CWT, °F (°C) Location Test Hours Properties	CM-1 550 (288)		CM-2 550 (288)		CM-3 625 (329)	
	Sump 69	Ring Zone 69	Sump 63	Ring Zone 63	Sump 53	Ring Zone 53
K. Vis., at 100°C, cSt	295.2	8.28	121.9	8.01	143.7	9.92
K. Vis., at 100°C, % Increase	1840	-46	701	-47	845	-35
TAN	12.3	7.7	7.8	IS*	12.3	9.9
TAN Change	+8.8	+4.2	+4.3	--	+8.8	+6.4
TBN	38	3.6	26.5	IS	31.1	5.2
TBN Change	+24	-10.4	+12.5	--	+17.1	-8.8
Sulfur, wt%	0.40	0.32	0.40	0.23	0.45	0.31
Nitrogen, wt%	0.231	0.183	0.252	ND**	0.207	0.163
Elements, ppm						
Ca	6400	1900	5300	300	1020	1900
Ba	NIL	NIL	6	NIL	NIL	NIL
Zn	2100	1100	2063	198	5500	1500
P	4000	2000	1667	315	4100	1300
Fe	104	53	57	10	251	49
Cr	3	4	2	<1	7	1
Pb	15	14	6	<1	16	6
Cu	8	201	4	3	11	21
Sn	6	2	<15	8	13	2
Al	27	7	12	<1	58	10
TGA Soot, wt%	2.9	1.3	1	0.6	ND	ND
Coagulated Insolubles						
C5	0.45	1.86	0.18	0.69	1.38	2.32
Toluene	0.43	1.47	0.16	0.47	1.30	1.87
GCBPD, °C at wt% off						
1	369	274	327	276	340	284
5	425	361	424	374	417	389
10	431	404	433	415	424	416
20	445	414	469	426	439	426
30	476	425	481	436	470	438
40	483	447	488	464	478	464
50	489	463	500	474	489	473
60	496	470	529	479	512	478
70	513	478	597	486	599	487
80	537	491	>600	497	>600	500
90	>600	528	>600	528	>600	538
95	>600	>600	>600	558	>600	>600
Residue, wt%, 600°C	12.0	5.2	30.6	3.1	30.5	6.0

* IS = Insufficient Sample.

** ND = Not Determined.

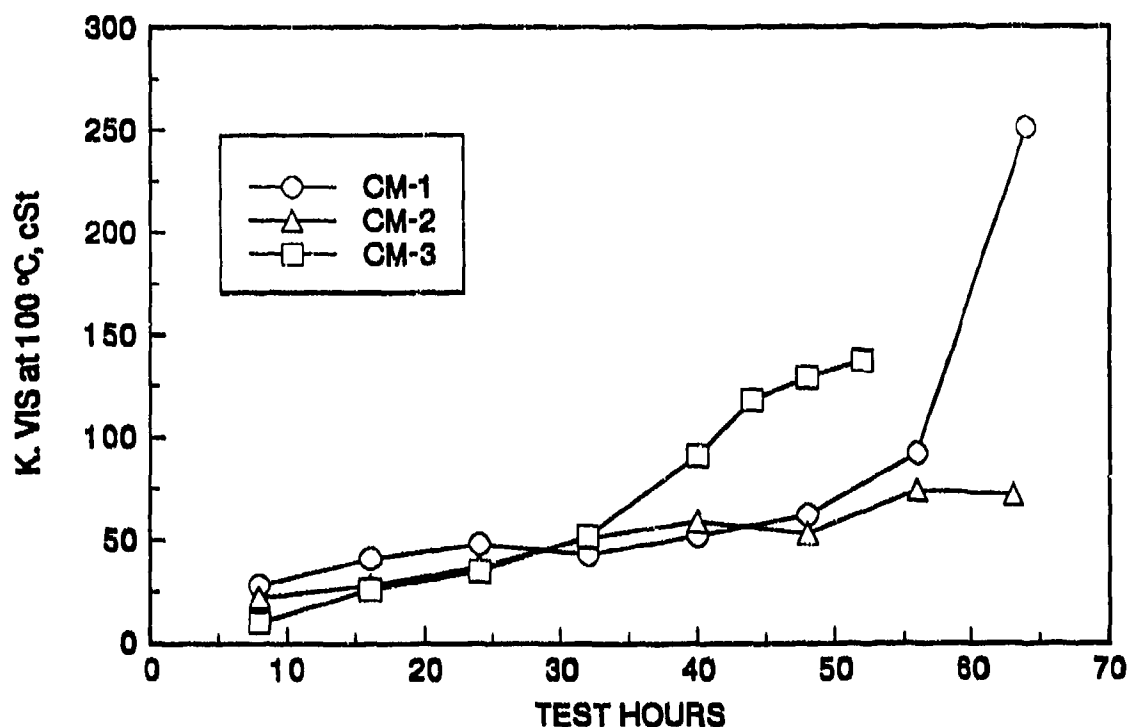


Figure 9. Viscosity at test hours (Oil CM)

Test CM-3 at 625°F (329°C) CWT was stopped at 53 test hours with excessive viscosity increase, TBN concentration (31.1), and 251 ppm iron in the used oil. Medium distress on the No. 2 compression ring accounted for the increased used oil iron content.

The RZOs from these tests (TABLE 18) had quite different properties than the used sump oil. In all three tests, RZO viscosity was reduced 35 to 47 percent from new oil viscosity, and RZO did not have the buildup of additive elements and TBN experienced in the sump oil. The GCBPD of the RZOs revealed a more volatile oil than the sump oil. This increased volatility could have been caused by some fuel dilution of the RZO or by thermal cracking of the base stock materials by the high temperature of the ring zone. Overall, Oil CM had inadequate viscosity increase control when operated at CWTs of 550°F or greater.

4. Oils D, E, and F

Oils D, E, and F were all evaluated at 631° to 633°F (333°C) CWT early in the project before the RZO collection concept was used. The properties of Oils D, E, and F are shown in TABLE 19, and the summarized results are presented in TABLE 20. Fig. 10 presents the plots of oil viscosity with test hours for Tests D-1, E-1, and F-1. The results for each oil are discussed in the following paragraphs.

Oil D is a commercially available synthetic oil (SAE 50 grade) that contained a base stock blend of diester and polyalphaolefin. Oil D was formulated for use as a multipurpose manual transmission fluid and diesel engine oil. While this oil contained no zinc antiwear/antioxidant additive, the manufacturer claimed API service classification CD, and it contains a heavy concentration of barium detergent-dispersant additive (1.48 wt% Ba). Test D-1 was run at an average CWT of 633°F (333°C), and completed 49 test hours. At the end-of-test, the oil had severely thickened, and the front main crankshaft bearing was damaged because of several low oil pressure startups, which resulted from the oil being too viscous to provide adequate flow. The barium additive was concentrated (4.91 wt%) in the used oil; however, the TBN did not increase. From Fig. 10 (viscosity versus test hours), Oil D had an expected useful life based on viscosity increase of less than 10 hours at 633°F CWT. Overall, Oil D had inadequate high-temperature performance.

Oil E is a commercially available synthetic diester/PAO diesel engine oil (API service classification CD) with SAE 10W-30 viscosity. This oil completed 50 hours at an average cylinder wall temperature of 633°F (333°C) when the test was terminated due to high oil viscosity (343 cSt at 100°C) and apparent top ring hot sticking; however, both the top and second compression rings were free when the engine was rated. Based on the viscosity increase plot in Fig. 10, Oil E did not reach the arbitrary limit of 50 cSt at 100°C until 40 test hours. In addition, the used oil TAN increased to 13.9, which indicates oil decomposition and oxidation. Overall, Oil E had inadequate performance for long-term use in low-heat rejection diesel engines operating at >600°F (316°C) CWT.

TABLE 19. Properties of Oils D, E, and F

Oil ID Properties	D	E	F
K. Vis, cSt, at 40°C	133.29	63.31	102.57
100°C	17.81	10.73	16.08
Viscosity Index	148	161	168
Flash Point, °C	227	221	227
Gravity, °API	22.8	ND*	ND
Sulfated Ash, wt%	2.38	1.96	1.01
TAN	1.6	3.8	3.0
TBN	10.6	13.2	7.0
Elements, wt%			
Ba	1.48	NIL	NIL
Ca	NIL	0.46	0.11
Mg	NIL	0.001	0.06
Zn	NIL	0.10	0.16
P	0.09	0.09	0.11
S	0.12	0.57	0.38
N	ND	0.061	ND
GCBPD, °C at wt% off			
1	308	245	279
5	368	375	368
10	407	406	396
20	437	422	444
30	446	436	474
40	451	452	481
50	459	459	486
60	471	467	490
70	497	473	496
80	>600	482	512
90	>600	506	564
Residue, wt%, 600°C	21.4	0	8.4

* ND = Not Determined.

TABLE 20. Summarized Test Results: Oils D, E, and F

Test No.	<u>D-1</u>	<u>E-1</u>	<u>F-1</u>
<u>Operating Conditions</u>			
Avg Cyl Wall Temp., °F (°C)	633 (334)	631 (333)	633 (334)
Min Avg CWT, °F (°C)	602 (317)	605 (318)	588 (309)
Max Avg CWT, °F (°C)	700 (371)	686 (363)	675 (357)
Gallery Oil Temp., °F (°C)	270 (132)	271 (133)	270 (132)
Speed, rpm	2000	2000	2000
Torque, ft-lb	23	25	26
Oil Consumption, lb/hr	0.85	0.69	0.70
<u>Results</u>			
Test Hours	48	50	50
Compression Ring Sticking			
Top	Free	Free	Free
Second	100% CS*	Free	35% CS
Third	Sluggish	100% CS	20% CS
Deposits			
Piston WTD	310	390	353
Piston Skirt Demerits			
Thrust	2.0	3.9	2.8
Antithrust	1.5	2.0	1.8
<u>Used Lubricant Properties</u>			
K. Vis, at 40°C, cSt	8557.59	10,855	2465.8
K. Vis, at 40°C, % Increase	6320	17046	2304
K. Vis, at 100°C, cSt	358.79	342.90	159.0
K. Vis, at 100°C, % Increase	1915	3095	889
TAN	1.0	13.9	10.8
TAN Change	-0.6	+10.1	+7.8
TBN	11.2	17.9	11.3
TBN Change	+0.6	+4.7	+4.3
Wear Metals, ppm			
Fe	37	45	72
Cu	<10	<10	<10
Pb	<60	<60	<60
Insolubles, wt%			
Pentane, B	0.38	1.23	0.52
Toluene, B	0.36	1.17	0.47
Elements, wt%			
Ca	NIL	3.88	0.42
Zn	NIL	1.10	0.46
P	0.29	0.53	0.33
Ba	4.91	NIL	NIL

* CS = Cold Stuck.

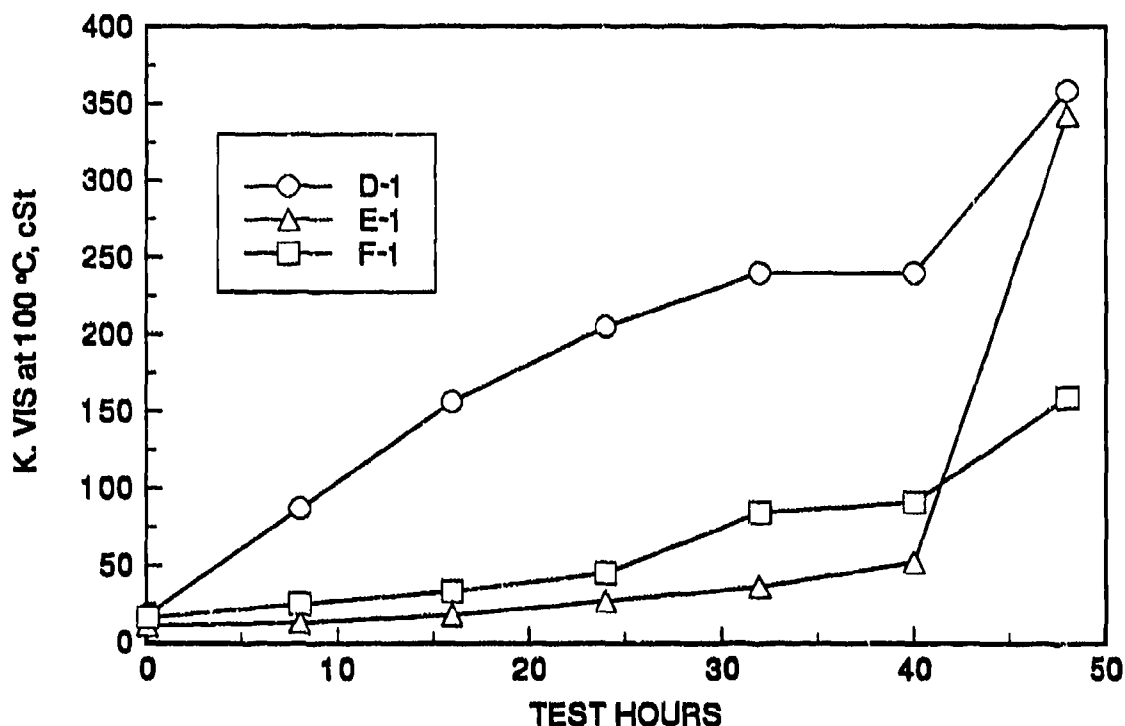


Figure 10. Viscosity at test hours (Oils D, E, and F)

Oil F was also a diester/PAO-based product and was SAE grade 15W-40, API service classifications SF/CD. A calcium/magnesium detergent-dispersant additive system was used. Oil F completed the scheduled 50 test hours at 633°F (333°C) CWT, and experienced substantial oil thickening (K. Vis = 159 cSt at 100°C), and the TAN increased to 10.8. As shown in Fig. 10, Oil F reached the 50 cSt at 100°C point at around 25 hours; however, it did not experience a sharp viscosity increase breakpoint. Overall, Oil F was intermediate in high-temperature performance compared to Oils D and E, but not satisfactory for long-term use in an LHR engine.

5. Oils G Through M

The next series of six oils (G, H, I, J, K, and L) evaluated in the VM diesel engine were all synthetic gas turbine engine oils, which met the requirements of MIL-L-23699. Oil G is a well-known widely used commercial product. TABLE 21 presents the new and used oil properties,

TABLE 21. Results With Oil G

Test No.	G-1			G-2		G-3	
CWT, °F (°C)	470 (243)			550 (288)		625 (329)	
Test Hours	At 100 Hr			At 100 Hr		At 77 Hr	
Oil ID	New Oil	Sump Oil	Ring Zone Oil	Sump Oil	Ring Zone Oil	Sump Oil	Ring Zone Oil
							58 to 69 Hr
<u>Oil Properties</u>							
K. Vis, at 100°C, cSt	5.36	6.36	6.53	9.64	10.27	12.50	7.03
K. Vis, at 100°C, % Increase	--	19	22	80	92	133	31
TAN	0.03	0.09	0.07	0.49	3.08	0.53	2.01
% N	0.110	0.154	0.165	0.328	0.356	0.421	0.452
% P	0.15	0.09	0.09	0.14	0.15	0.14	0.14
% S	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	0.01
TBN	<0.01	ND*	ND	ND	ND	ND	ND
<u>GCBPD, °C at wt% off</u>							
1	408	414	402	381	335	404	358
5	427	440	434	435	428	429	433
10	435	448	441	445	438	437	443
20	446	459	451	457	450	450	456
30	453	467	460	469	461	460	469
40	462	475	467	478	472	470	480
50	470	484	479	488	484	482	494
60	481	493	488	506	503	501	525
70	492	508	511	545	544	547	580
80	513	553	563	>600	>600	>600	>600
90	>600	>600	>600	>600	>600	>600	>600
Residue, wt%, 600°C	10.9	13.5	16.4	22.0	21.2	22.2	27.8
Fe, ppm	<1	24	25	32	22	127	111
Cu, ppm	<1	7	6	7	4	8	21
Pb, ppm	<1	2	3	<1	<1	<1	1
<u>Engine Rating at End-of-Test</u>							
	G-1		G-2		G-3		
Piston Deposits, WTD	268		345		461		
Ring Sticking, Top Compression	Free		Free		Free		
No. 2 Compression	Free		20% Cold Stuck		100% Cold Stuck		
No. 3 Compression	Free		Free		Free		
Oil Ring	Free		Sluggish		Free		
Oil Ring Plugging, %	5		80		93		
Piston Varnish (Demerits)							
Thrust Side	7.5		7.8		7.1		
Antithrust Side	6.8		7.6		6.1		
<u>Engine Operating Conditions</u>							
Avg Cyl Wall Temp., °F (°C)	470 (243)		550 (288)		625 (329)		
Min Avg CWT, °F (°C)	423 (217)		494 (251)		562 (294)		
Max Avg CWT, °F (°C)	546 (286)		650 (343)		711 (377)		
Gallery Oil Temp., °F (°C)	270 (132)		269 (132)		269 (132)		
Speed, rpm	2008		2011		2011		
Torque, ft-lb	27		28		28		
Oil Consumption, lb/hr	0.26		0.47		0.71		

* ND = Not Determined.

and VM engine test operation and results summary for Oil G, which was evaluated at 470°, 550°, and 625°F (243°, 288°, and 329°C) CWT. Fig. 11 shows the viscosity increase versus test hours for these three tests, while Fig. 12 shows the used oil iron content with test hours. At 470° and 550°F (243° and 288°C) CWT, Oil G completed 100 test hours without excessive viscosity increase or used oil iron accumulation. At 625°F (329°C) CWT, Test G-3 was stopped at 77 hours because of a sudden increase in used oil iron content, which was subsequently found to be caused by a broken oil ring expander. Oil consumption for these three tests is shown in Fig. 13, and end-of-test piston deposits (WTD) are shown in Fig. 14. The effect of increasing CWT was well quantified. In going from 470° to 625°F (243° to 329°C) CWT, oil consumption increased by 2.7 times and piston WTD increased by 1.7 times. RZO was collected throughout Tests G-1 and G-2 and for 58 to 62 hours during Test G-3. At 470°F (243°C) CWT (Test G-1), RZO properties were very similar to the used sump oil. For Test G-2 [550°F (288°C) CWT], RZO had higher TAN and slightly higher viscosity, which indicated slightly greater oxidation than the sump oil. Test G-3 RZO had limited degradation because of the relatively short collection time. Overall, Oil G had acceptable performance at 470°F (243°C) CWT, marginal performance at 550°F (288°C) CWT because of ring deposits and sticking, and unacceptable performance at 625°F (329°C) CWT because of ring deposits and sticking. The broken oil ring expander may have been related to oil ring plugging.

Oil H (MIL-L-23699) was evaluated at 462°F (241°C) CWT (Test H-1) and 630°F (332°C) CWT (Test H-2). TABLE 22 presents the new and used oil properties, operational, and results summaries for Oil H. Test H-1 was stopped at 25 hours because of high engine blowby, which increased steadily throughout the test. Sump oil and RZO both had mild degradation at EOT, and the piston deposits were relatively low. The blowby increase is believed to be the result of intermittent ring sticking during operation. Test H-2 at 630°F (332°C) CWT was stopped at 12 hours because of extremely high blowby. The No. 2 and 3 compression rings and the oil ring were all 100 percent cold stuck. Used oil iron content was high (218 ppm), while other oil degradation was not severe. Overall, Oil H was not suitable for use at 630°F CWT. Even at 462°F CWT, the VM engine evaluation had to be stopped well in advance of many other candidate HTLs.

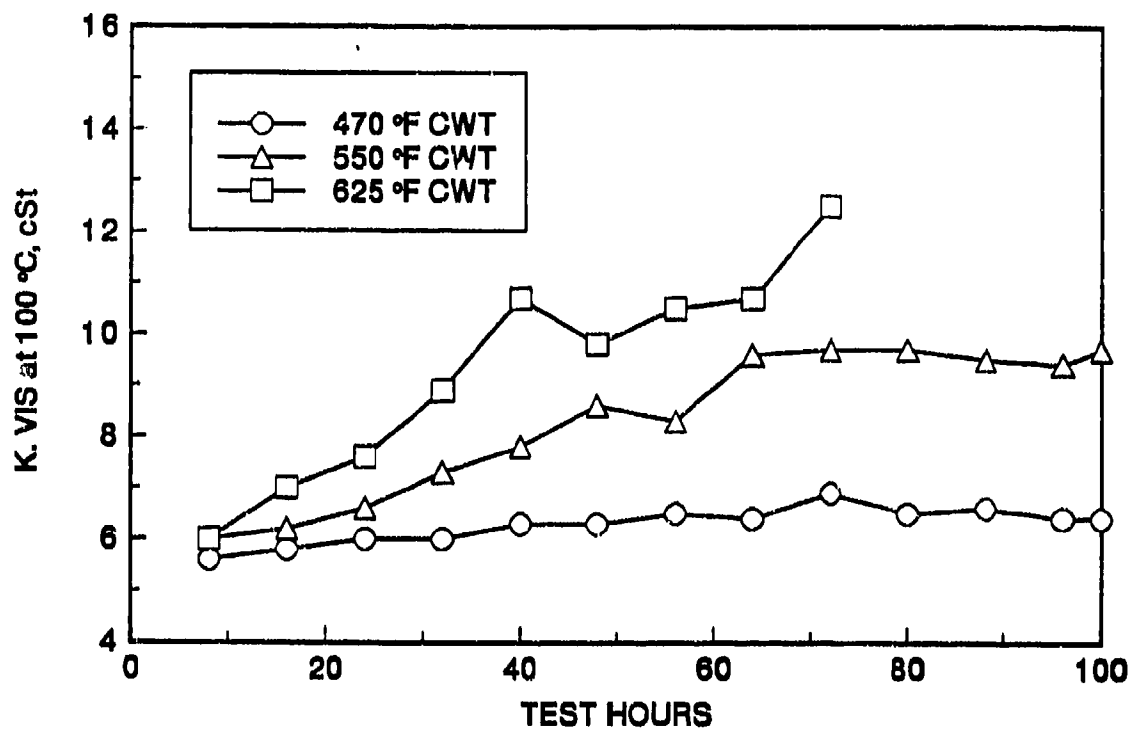


Figure 11. Viscosity increase (Oil G)

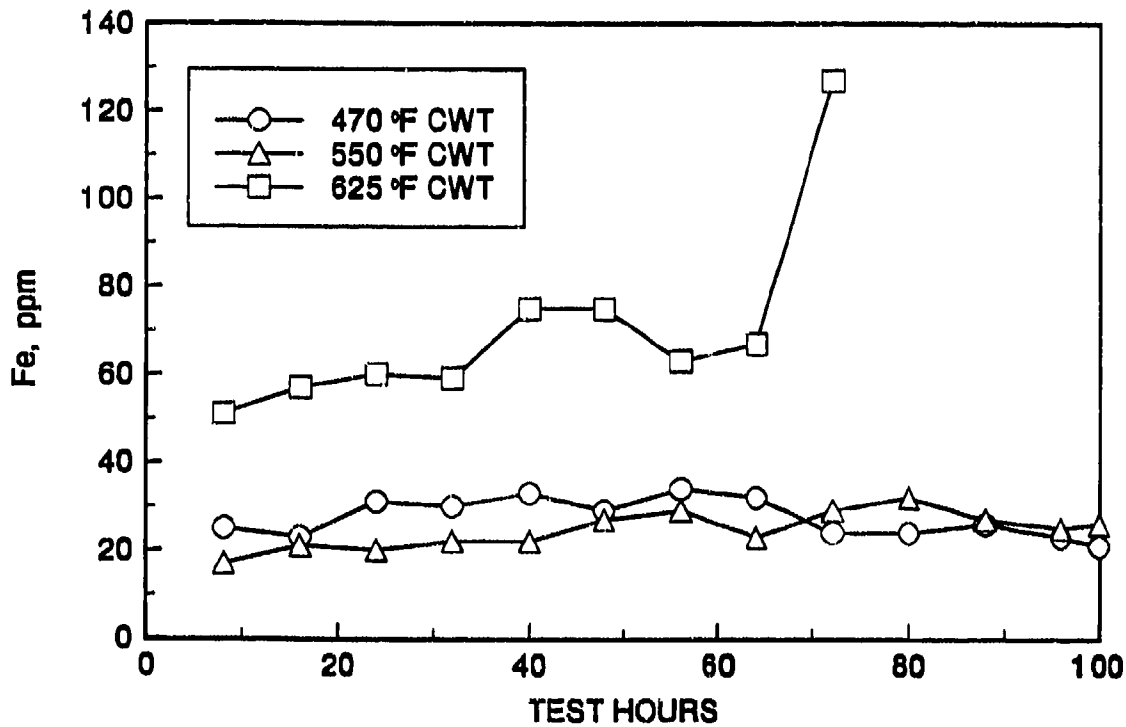


Figure 12. Used oil iron content (Oil G)

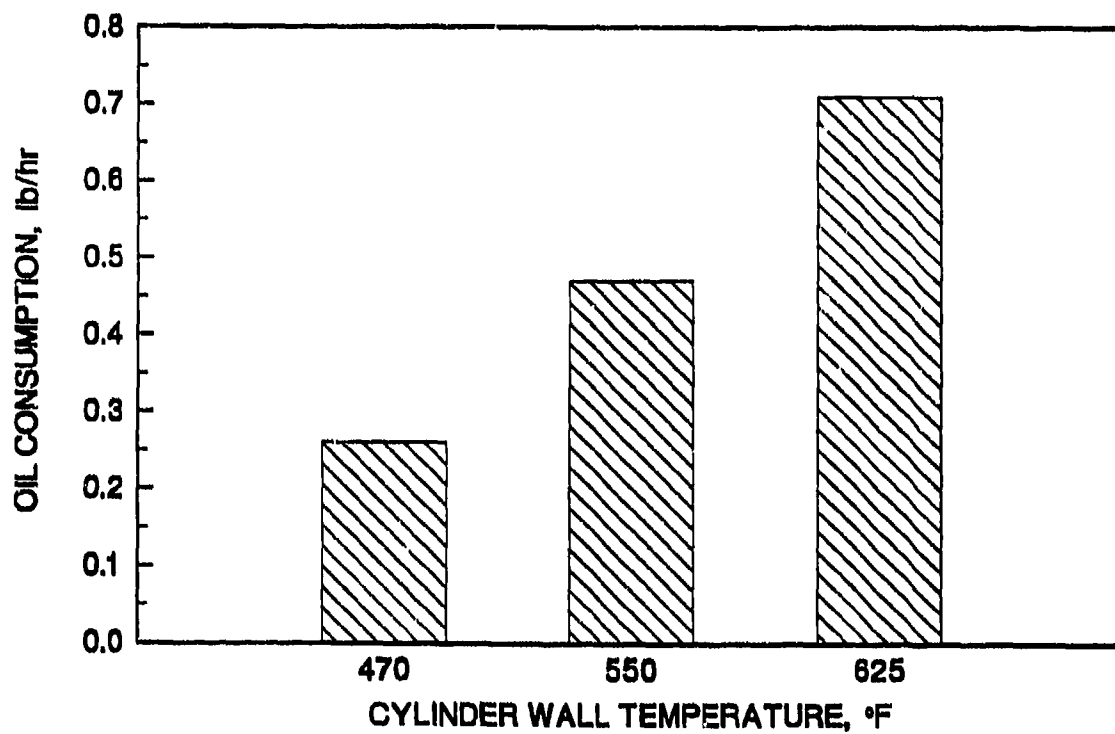


Figure 13. Oil consumption (Oil G)

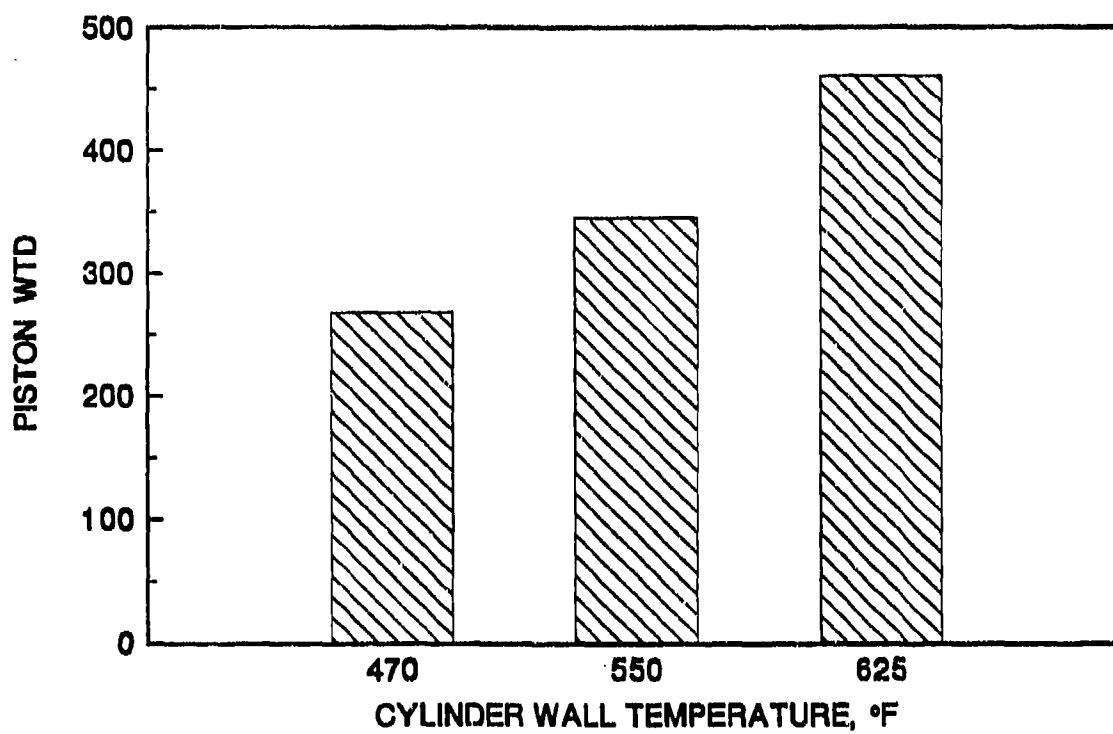


Figure 14. Piston WTD (Oil G)

TABLE 22. Results With Oil H

Test No. CWT, °F (°C) Test Hours	H-1 462 (239) At 25 Hr			H-2 630 (332) At 12 Hr	
Oil ID	New Oil	Sump Oil	Ring Zone Oil	Sump Oil	Ring Zone Oil
<u>Oil Properties</u>					
K. Vis, at 100°C, cSt	5.04	5.35	5.82	5.42	5.70
K. Vis, at 100°C, % Increase	--	6	15	8	13
TAN	0.07	0.08	1.11	0.73	3.27
% N	0.08	0.07	0.09	0.09	0.124
% P	0.04	0.10	0.09	0.10	0.09
% S	0.01	<0.01	0.01	0.04	<0.01
<u>GCRPD, °C at wt % off</u>					
1	359	364	380	349	336
5	413	417	423	413	417
10	420	424	439	416	424
20	439	441	448	436	445
30	445	448	457	442	451
40	453	456	471	457	469
50	468	471	478	466	477
60	475	478	490	475	488
70	486	492	505	496	509
80	506	509	532	523	543
90	543	552	>600	>600	>600
Residue, wt%, 600°C	4.6	6.4	12.2	11.0	12.9
Fe, ppm	1	38	50	218	182
Cu, ppm	1	1	2	9	16
Pb, ppm	1	2	1	2	<1
<u>Engine Rating at End-of-Test</u>					
	H-1			H-2	
Piston Deposits, WTD	140			380	
Ring Sticking, Top Compression	Free			Free	
No. 2 Compression	Free			100% Cold Stuck	
No. 3 Compression	Free			100% Cold Stuck	
Oil Ring	Free			100% Cold Stuck	
Oil Ring Plugging, %	<1			<1	
Piston Varnish (Demerits)					
Thrust Side	4.8			7.8	
Antithrust Side	3.8			7.4	
<u>Engine Operating Conditions</u>					
Avg Cyl Wall Temp., °F (°C)	462 (239)			630 (332)	
Min Avg CWT, °F (°C)	429 (221)			553 (289)	
Max Avg CWT, °F (°C)	531 (277)			727 (386)	
Gallery Oil Temp., °F (°C)	260 (127)			269 (132)	
Speed, rpm	2004			2023	
Torque, ft-lb	27			26	
Oil Consumption, lb/hr	0.659			0.698	

Oils I and J were also MIL-L-23699 gas turbine engine oils that were evaluated in the VM engine at 470° and 464°F (243° and 240°C) CWT, respectively. The new and used oil properties, and operational and results summaries are presented in TABLE 23. Evaluation of Oil I was stopped at 21 hours because of high blowby. Oil consumption was very high (1.08 lb/hr) due in part to leakage at the rear main seal. The ring zone oil and slight viscosity and TAN increases compared to the new oil and an increase in GCBPD residue were observed. Due to the large amount of makeup oil added because of very high oil consumption during this test, the sump used oil had nearly the same properties as the new oil. Overall, Oil I did not have adequate performance in the VM high-temperature diesel engine.

Oil J completed 24 hours when the test was stopped due to high blowby and extremely high oil consumption (1.88 lb/hr). Oil leakage at the rear main seal contributed substantially to the excessive oil consumption. The ring zone oil had the following characteristics as compared to the new oil: slightly increased viscosity, TAN and Gas Chromatography Boiling Point Distribution (GCBPD) residue. The sump used oil had very low wear metals content and had nearly the same properties as the new oil due to the excessive amount of makeup oil added during the test. As with Oil I, Oil J was not suitable for high-temperature diesel engine operation.

Oils K and L were the final MIL-L-23699 gas turbine engine oils evaluated in the high-temperature VM diesel engine. TABLE 24 contains the new and used oil properties, and operational and results summaries for Oils K and L. Oil K was evaluated in the VM engine at 545°F (285°C) CWT. The test was stopped at 10 hours because of extremely high used oil iron content. The engine was disassembled, and piston scuffing (15 percent) was observed. Oil K was considered as being unacceptable for high-temperature diesel engine operation. Oil L was evaluated at 550° and 615°F (288° to 324°C) CWT. Test L-1 (550°F CWT) completed 100 hours. End-of-test engine inspection revealed the following: compression rings 2 and 3 were 100 percent cold stuck, and the oil ring was also 65 percent plugged. Ring zone oil was slightly more degraded than the sump oil (viscosity increase, TAN). Test L-2 was conducted at 615°F (324°C) CWT, and was stopped at 11 hours because of very high used oil iron content

TABLE 23. Results With Oils I and J

Test No.	I-1			J-1		
CWT, °F (°C)	470 (243)			464 (240)		
Test Hours	At 21 Hr			At 24 Hr		
Oil ID	New Oil	Sump Oil	Ring Zone Oil	New Oil	Sump Oil	Ring Zone Oil
<u>Oil Properties</u>						
K. Vis, at 100°C, cSt	4.94	5.04	5.38	5.10	5.13	5.55
K. Vis, at 100°C, % Increase	--	2	9	--	0.6	9
TAN	0.01	0.09	0.77	<0.01	0.10	1.40
% N	0.106	0.09	0.09	<0.01	0.13	0.05
% P	0.13	0.13	0.10	0.129	0.115	0.154
% S	<0.01	<0.01	<0.01	0.05	0.07	0.07
<u>GCBPD, °C at wt% off</u>						
1	363	363	402	396	399	341
5	423	422	428	431	432	431
10	432	431	437	436	438	438
20	444	444	447	444	445	446
30	451	451	456	451	452	454
40	460	460	465	458	460	462
50	468	468	474	466	467	470
60	478	478	484	475	476	480
70	489	489	496	485	486	492
80	506	507	519	501	503	519
90	543	547	569	551	559	>600
Residue, wt%, 600°C	4.4	5.2	8.0	6.2	7.5	11.7
Fe, ppm	<1	2	23	1	14	18
Cu, ppm	<1	<1	2	1	1	2
Pb, ppm	2	<1	1	1	1	2
<u>Engine Rating at End-of-Test</u>						
	I-1			J-1		
Piston Deposits, WTD	210			139		
Ring Sticking, Top Compression	Free			Free		
No. 2 Compression	Free			Free		
No. 3 Compression	Free			Free		
Oil Ring	Free			Free		
Oil Ring Plugging, %	<1			<1		
Piston Varnish (Demerits)						
Thrust Side	4.0			3.5		
Anti-thrust Side	2.8			3.0		
<u>Engine Operating Conditions</u>						
Avg Cyl Wall Temp., °F (°C)	470 (243)			464 (240)		
Min Avg CWT, °F (°C)	424 (218)			413 (217)		
Max Avg CWT, °F (°C)	561 (294)			544 (284)		
Gallery Oil Temp., °F (°C)	266 (130)			267 (131)		
Speed, rpm	2008			2008		
Torque, ft-lb	26			26		
Oil Consumption, lb/hr	1.08			1.88		

TABLE 24. Results With Oils K and L

Test No. CWT, °F (°C) Test Hours Oil ID	K-1 545 (285) At 10 Hr		L-1 550 (288) At 100 Hr			L-2 615 (324) At 11 Hr
	New Oil	Sump Oil	New Oil	Sump Oil	Ring Zone Oil	Sump Oil
Oil Properties						
K. Vis, at 100°C, cSt	5.05	5.77	5.20	6.98	7.98	6.09
K. Vis, at 100°C, % Increase	--	14	--	34	53	17
TAN	0.05	ND*	0.15	0.94	2.07	ND
% N	0.094	ND	0.132	0.128	0.186	ND
% P	0.10	ND	0.03	0.04	0.04	ND
% S	<0.01	ND	<0.01	<0.01	<0.01	ND
GCBPD, °C at wt% off						
1	359	ND	412	418	387	ND
5	411	ND	430	438	442	ND
10	418	ND	434	446	451	ND
20	435	ND	444	457	463	ND
30	444	ND	451	468	474	ND
40	451	ND	460	478	485	ND
50	463	ND	467	491	500	ND
60	474	ND	477	513	531	ND
70	483	ND	488	553	565	ND
80	505	ND	508	>600	>600	ND
90	540	ND	574	>600	>600	ND
Residue, wt%, 600°C	5.5	ND	8.9	23.4	25.5	ND
Fe, ppm	<1	1250	<1	253	258	551
Cu, ppm	<1	<10	<1	9	22	<10
Pb, ppm	<1	<60	<1	<1	<1	<10
Engine Rating at End-of-Test						
	K-1		L-1		L-2	
Piston Deposits, WTD	166		370		365	
Ring Sticking, Top Compression	Free		Free		Free	
No. 2 Compression	Free		100% Cold Stuck		100% Cold Stuck	
No. 3 Compression	Free		100% Cold Stuck		100% Cold Stuck	
Oil Ring	Free		100% Cold Stuck		100% Cold Stuck	
Oil Ring Plugging, %	<1		65		9	
Piston Varnish (Demerits)						
Thrust Side	2.8		5.2		5.0	
Antithrust Side	2.3		4.2		3.7	
Engine Operating Conditions						
Avg Cyl Wall Temp., °F (°C)	545 (285)		550 (288)		615 (324)	
Min Avg CWT, °F (°C)	484 (251)		504 (262)		524 (273)	
Max Avg CWT, °F (°C)	637 (356)		646 (341)		754 (401)	
Gallery Oil Temp., °F (°C)	268 (131)		270 (132)		269 (132)	
Speed, rpm	2037		2024		2028	
Torque, ft-lb	26		25		27	
Oil Consumption, lb/hr	0.265		0.402		0.877	

* ND = Not Determined.

(551 ppm). Engine inspection revealed 80 percent liner scuffing on the thrust side, and 100 percent cold stuck Nos. 2 and 3 compression ring and the oil ring. Overall, Oil L had marginally acceptable VM engine performance at 550°F (288°C) CWT, but was not suitable for use at 615°F (324°C) CWT.

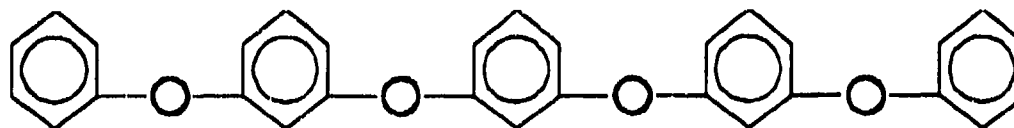
The high-temperature diesel engine performance of the various gas turbine engine oils in terms of test length versus CWT is summarized in TABLE 25. Oil G had the best overall performance based on test duration time.

TABLE 25. Summary of TEO Tests in the HT VM Diesel Engine

Oil CWT, °F (°C)	Test, Hr					
	<u>G</u>	<u>H</u>	<u>I</u>	<u>J</u>	<u>K</u>	<u>L</u>
470 (243)	100	25	21	24	--*	--
550 (288)	100	--	--	--	10	100
630 (332)	77	12	--	--	--	11

* -- = Not Applicable.

The final oil evaluated in the high-temperature VM diesel engine was a polyphenylether base stock without additives (Oil M). The polyphenylether had the following structure:



5P-4E

TABLE 26 presents the new and used oil properties, and operational and results summaries for Oil M. Because of its high pour point, Oil M was preheated prior to addition to the engine. The test completed 16 hours at 550°F (288°C) CWT, and was stopped due to high blowby. The engine was disassembled, and the piston rings and grooves were cleaned and the test was

TABLE 26. Results With Oil M

Test No.	<u>M-1</u>
<u>Operating Conditions</u>	
Avg Cyl Wall Temp, °F (°C)	547 (286)
Min Avg CWT, °F (°C)	518 (270)
Max Avg CWT, °F (°C)	604 (318)
Gallery Oil Temp., °F (°C)	268 (131)
Speed, rpm	2036
Torque, ft-lb	26
Oil Consumption, lb/hr	0.42

Results

Test Hours	16	21
Compression Ring Sticking		
Top	Free	Free
Second	Free	Free
Third	Free	Free
Deposits		
Piston WTD	195	200
Piston Skirt Demerits		
Thrust	7.5	4.5
Antithrust	6.5	4.2
Other Distress	Ring Face Distress High Blowby	

Test Hr Location	<u>0 New</u>	<u>21 Sump</u>	<u>21 Ring Zone</u>
<u>Oil Properties</u>			
K. Vis, at 40°C, cSt	290.67	306.96	ND*
K. Vis, at 100°C, cSt	12.72	13.58	11.97
Viscosity Index	BSOM**	BSOM	BSOM
TAN	0.25	0.54	0.93
TBN, D 664	0.12	0.87	0.51
Sulfur, wt%	NIL	NIL	NIL
<u>Elements, ppm by ICP</u>			
Ca	<1	172	154
Ba	1	12	3
Mg	1	172	139
Zn	4	86	73
P	4	126	99
B	<1	16	21
Fe	<1	182	41
Cr	<1	3	<1
Pb	<1	3	<1
Cu	<1	4	1
Sn	370	536	240
Al	<1	42	3

* ND = Not Determined.

** BSOM = Beyond Scope of Method.

resumed. The test was stopped at 21 hours due to a sudden increase in iron wear metal. The top and No. 2 compression rings had moderate distress. Fig. 15 shows the unusual piston deposits with Oil M. The used oils had virtually no increase in viscosity or TAN. The oil tin content was high from the start of the test due to contamination of the 5P-4E material. The trace additive elements in the used oils resulted from break-in oil hang-up in the engine. Overall, Oil M was not satisfactory due to engine deposits.

A comparison of key high-temperature VM oil test results for HTL evaluations at 470°F (243°C), 550°F (288°C), 600°F (316°C), 630°F (332°C), and 650°F (343°C) CWT is presented in TABLE 27. At 470°F (243°C) CWT, Oils G and A had the best overall performance, while Oils H, I, and J were not acceptable. At 550°F (288°C) CWT, Oil G had the best overall performance. Oils K, L, and M all had excessive used oil iron content. At 600°F (316°C) CWT, Oil B had the best performance because of longer test duration. At 630°F (332°C) CWT, Oil G had the longest test duration and best overall performance. Oils M, H, and L had excessive used oil iron contents. At 650°F (343°C) CWT, both Oils A and B had poor performance because of ring sticking and high blowby. Overall Oil G had the best high-temperature performance at CWTs of $\geq 630^\circ\text{F}$ (332°C). Oil B had the second best overall performance, while Oil A had the third best performance.

IV. BENCH TESTS FOR HTL SCREENING

A. Oxidation

An oxidation-corrosion bench test based on modifications of Method 5307 of FTMS-791C, "Corrosiveness and Oxidation Stability of Aircraft Turbine Engine Lubricants," (18) was used to investigate HTL oxidation. The major modification from Method 5307 was the use of cast iron, copper, lead, and aluminum metal specimens, which are representative of diesel engine components. Summarized information concerning the modified test is presented in TABLE 28. The initial evaluations were conducted at 450°F (232°C) for 48 hours. Summarized results are presented in TABLE 29, and the oil codes correspond to the oils discussed in Section III.

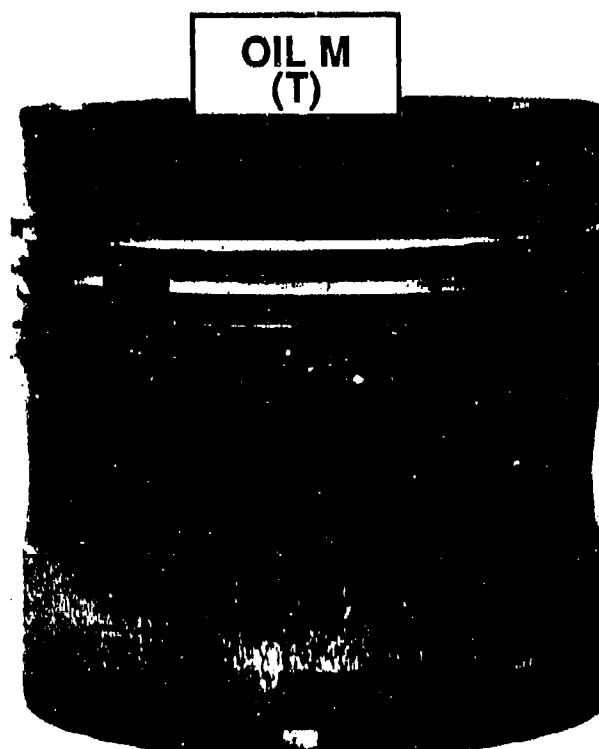
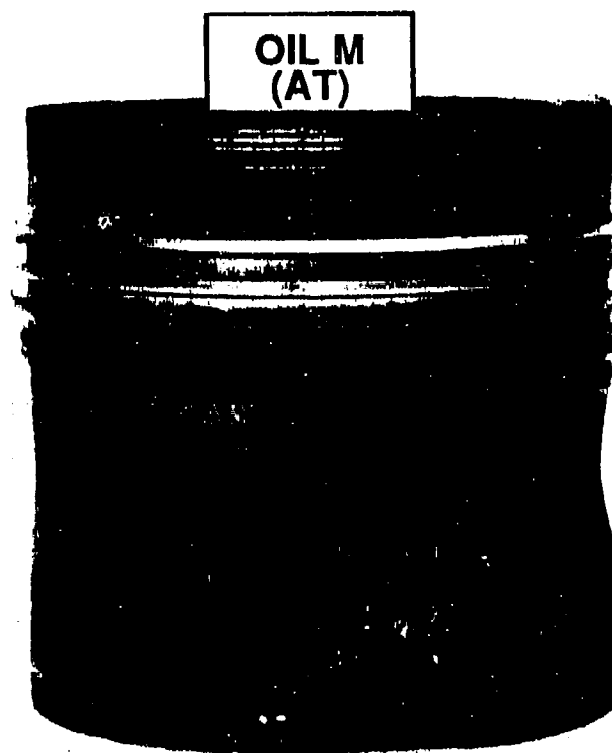


Figure 15. VM piston — Oil M, 21 hours

TABLE 27. Summary of HTL Evaluations at Fixed CWT

CWT	Test, Hr	Via Increase,	Oil Consumption, lb/hr	Fe, ppm	Ring Sticking		Piston WTD	Other
		K. Vis. at 100°C, %			2 CR	3 CR		
470°F (243°C)								
Oil A	163	59	0.18	59	NA*	NA	NA	
Oil B	89	31	0.38	25	100**	F***	214	
Oil G	100	19	0.26	24	F	F	268	
Oil H	25	6	0.66	38	F	F	140	High Blowby
Oil I	21	2	1.08	2	F	F	210	Oil Leaks, Blowby
Oil J	24	<1	1.88	14	F	F	139	Oil Leaks, Blowby
550°F (288°C)								
Oil B (B-3)	75	140	0.39	52	100	F	440	
Oil CM (CM-1)	69	1840	0.47	104	100	S†	290	
Oil G	100	80	0.47	32	20	F	345	
Oil K	10	NIL	0.27	1250	F	F	166	Piston Scuffing, High Fe
Oil L	100	34	0.40	253	100	100	370	High Fe
Oil M	21	7	0.42	182	F	F	200	High Blowby, High Fe
600°F (316°C)								
Oil A	50	32	0.99	15	100	100	497	
Oil B (B-5)	102	66	0.95	87	100	75	401	
Oil C	50	18	0.54	41	100	100	392	
630°F (332°C)								
Oil A	46	23	1.04	92	100	100	441	High Blowby
Oil B	41	49	0.89	62	100	100	424	High Blowby
Oil C	49	24	0.72	43	100	100	548	Tan Increase
Oil CM	53	845	0.71	251	100	100	298	High Wear, High Fe
Oil D	48	1915	0.85	37	100	S	310	
Oil E	50	3096	0.69	43	F	100	390	
Oil F	50	889	0.70	72	35	20	353	
Oil G	77	133	0.71	127	100	F	461	
Oil H	12	8	0.70	218	100	100	380	High Blowby, High Fe
Oil L	11	17	0.88	551	100	100	365	Liner Scuffing, High Fe
650°F (343°C)								
Oil A	19.5	69	0.41	40	100	F	302	High Blowby, Cleaned, 2 CR at 14 hr
Oil B	27	79	0.43	77	NA	NA	ND‡	Rings Stuck at 15 hr

* NA = Not Applicable.
** Value is percent cold stuck.
*** F = Free.
† S = Stuck.
‡ ND = Not Determined.

TABLE 28. Oxidation-Corrosion Test (FTM-5307 Modified)

- Oil, 200 mL
- Glassware Heated in Al Block
- Oxygen Flow, 10L/Hr
- Metal Specimens
 - Cast Iron
 - Copper
 - Lead
 - Aluminum
- Temperature: 450°F (232°C) or 600°F (316°C)
- Time: 48 hr (16-, 24-, 40-, 48-hr oil samples)
- Measure Change in:
 - Viscosity
 - TAN
 - Metal Weight

TABLE 29. Oxidation-Corrosion Test Results—450°F (232°C)/48 Hr

Oil Code	None	B	A	E	F
Lubricant Type	Petroleum	Petroleum	Polyol Ester	PAO/Diester	PAO/Diester
Results					
Vis Increase, %	1355	52	116	153	3300
Δ TAN	4.1	2.4	5.2	3.2	5.4
Oil Loss, %	5.8	5.8	6.6	8.0	8.8
Normalized Metal Wt Loss					
Cu	3.6	3.5	3.6	1.0	6.1
Pb	1.0	1.5	2.2	10.0	2.6
Al	0	0	0	0	0
Fe	5.6	6.9	8.8	1.0	6.0

Oils A, B, E, and F and another petroleum-based SAE 15W-40 oil were evaluated. Oil B had the least viscosity increase, while Oil F had extreme increase; however, none of the oils had excessive TAN buildup. The metal corrosion results are presented as relative to the best oil in this series. Severe leak-back was experienced with Oil E, and Oil A attacked the cast iron. Oil oxidation in this bench test at 450°F (232°C)/48 hours was compared with oil performance in the VM engine at >600°F (316°C) CWT, conditions at which Oils A, B, E, and F were run. In the VM engine, Oils A and B had similar viscosity increase performance, while Oil E had excessive (3000 percent) viscosity increase, and Oil F had a large increase (900 percent). In the 48-hour ox-cor test at 450°F (232°C), the order (best to worst) of oil oxidation performance was Oils B, C, E, and F. The bench test misordered the engine performance of Oils E and F. Additional ox-cor tests were conducted using the same methodology, but at 600°F (316°C) to determine lubricant oxidation at cylinder wall temperatures. An oil sample was taken at 2-hour intervals for a total of 8 hours to monitor viscosity and TAN changes. The summarized results for Oils A through F are presented in TABLE 30. Fig. 16 shows the plot of percent viscosity increase with test hours. Most of the oils appeared to first thermally crack and lose viscosity. Several oils then regained viscosity as oxidation and evaporation of light materials occurred. Because of the initial viscosity loss, these conditions were judged as being too severe to simulate overall ring zone and sump oil oxidation. Thermal cracking behavior of the bulk oil at 600°F (316°C)

TABLE 30. Oxidation-Corrosion Test Results—600°F (316°C)/8 Hr

Oil Code	A	B	C	D	E	F
Lubricant Type	Polyol Ester	Petroleum	Polyol/PAO	PAO/Diester	PAO/Diester	PAO/Diester
Test Hr	16	8	8	8	8	8
Results						
Δ Vis, %	ND at 2 Hr -4 at 4 Hr 9 at 8 Hr 50 at 16 Hr	-35 at 2 Hr -34 at 4 Hr -12 at 8 Hr	-37 at 2 Hr -36 at 4 Hr -28 at 8 Hr	-8 at 2 Hr -12 at 4 Hr 9 at 8 Hr	-34 at 2 Hr -35 at 4 Hr -32 at 8 Hr	ND at 2 Hr -45 at 4 Hr -16 at 8 Hr
Δ TAN	5.5	-0.6	+0.8	12.8	4.4	14.1
Oil Loss, %	15	6	12	14	11	12
Normalized Metal Wt Loss						
Cu	7	4	5	1	7	6
Pb	4	1	1	3	1	2

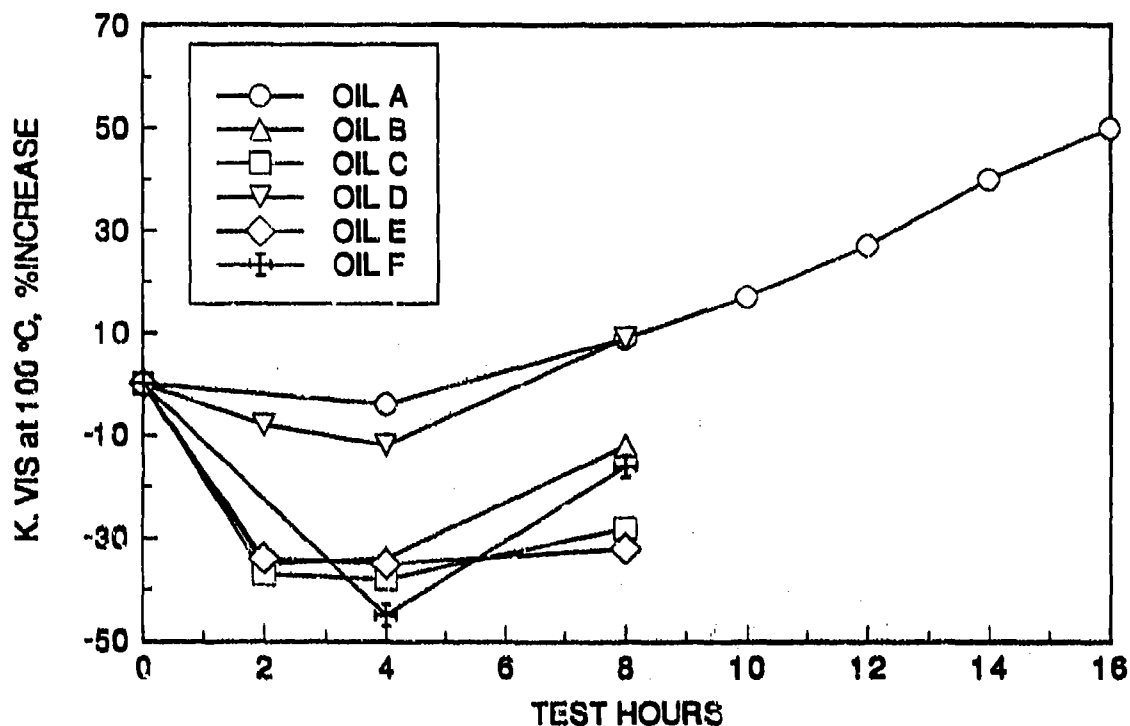


Figure 16. Oxidation-corrosion test at 600°F (316°C)

provided valuable insight into oil degradation in the HT diesel engine ring zone. Thin film oil in the ring zone area could experience even greater degradation.

B. Friction and Wear Characteristics

A Cameron-Plint friction and wear apparatus was obtained for friction and wear evaluations. A schematic of the rig is shown in Fig. 17. This rig allows friction and wear measurements to be made using a reciprocating wear piece, which is loaded and moved against a fixed wear piece. In the Cameron-Plint test apparatus, the following parameters are variables:

- Load
- Temperature
- Wear piece material and shape
- Fixed specimen heating rate
- Stroke
- Lubricant application
- Frequency
- Test duration.

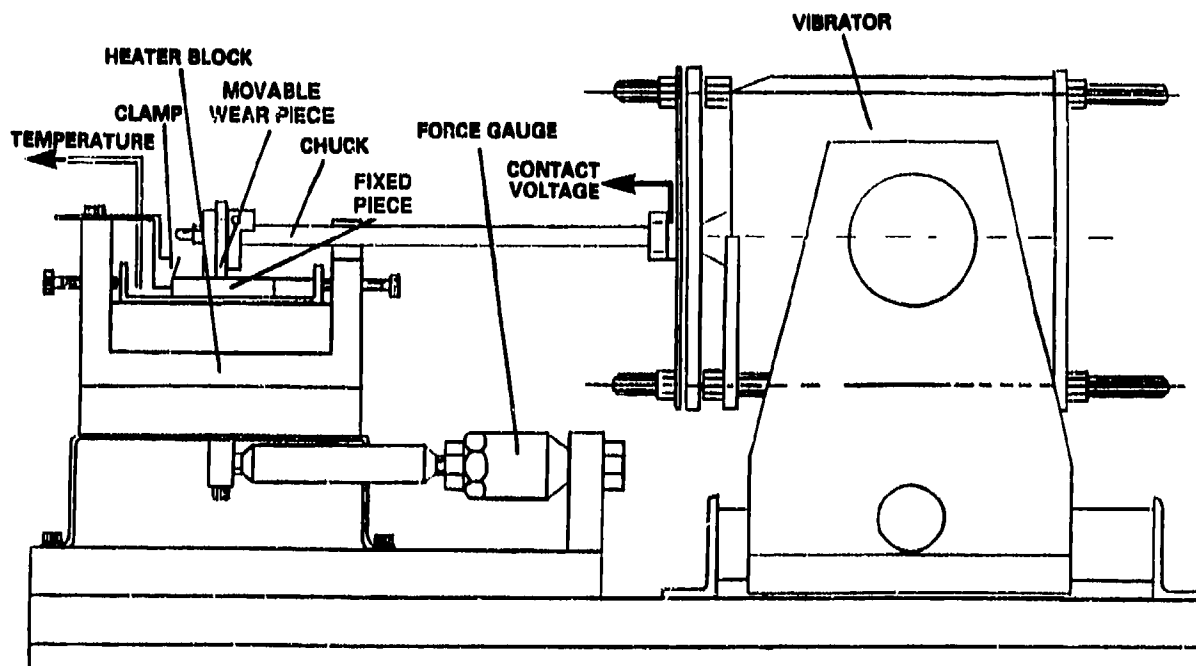


Figure 17. Schematic of Cameron-Plint apparatus

No standard operating condition exists for the Cameron-Plint rig; thus, conditions must be developed based on the objective of the experiment.

The objective of these experiments was to determine the effect of zinc and phosphate additives on friction. Tool steel specimens (T-15) were used as a pin sliding on flat for these determinations. The test matrix consisted of using petroleum, PAO, and ester-base stocks neat and with varying concentrations of alkyl zinc dithiophosphate (ZDTP) and tricresylphosphate (TCP). In addition, two 5-cSt gas turbine engine oils (TEO) were tested neat and with the ZDTP and TCP. The operating conditions and results of these tests are summarized in TABLE 31. The friction coefficient data show that, in the case of petroleum-based oils, there is an optimum requirement in the case of zinc additive, i.e., 0.1 wt% zinc has a deleterious effect on base-oil performance but 0.2 wt% zinc improves the overall friction coefficient considerably for the duration of the test. TCP in low concentration does not seem to affect the petroleum-based oils' friction coefficient. The ZDTP additive at both 0.1 wt% Zn and 0.2 wt% Zn levels improves the

TABLE 31. Effect of Zinc and Phosphate Additives on Friction in Plint Test

T-15 Tool Steel Specimens - Pin on Flat
1.5 Hours - Test Duration

Load - 200 N
Stroke - 2.31 mm at 40 Hz
Temp. - 120°C

Lubricant		Coefficient of Friction	
		Minimum	Maximum
None		0.3	0.46
PAO Base Stock	AL-12570-L	0.06	0.28
	AL-12570-L + 0.1 wt% Zn*	0.04	0.05
	AL-12570-L + 0.2 wt% Zn	0.04	0.05
	AL-12570-L + 5 wt% TAP**	0.09	0.36
	AL-12570-L + 15 wt% TAP	0.05	0.10
	AL-12570-L + 25 wt% TAP	0.04	0.07
Ester Base Stock	AL-6709-L	0.045	0.055
	AL-6709-L + 0.1 wt% Zn	0.05	0.35
	AL-6709-L + 0.2 wt% Zn	0.06	0.37
	AL-6709-L + 5 wt% TAP	0.07	0.395
	AL-6709-L + 15 wt% TAP	0.085	0.41
	AL-6709-L + 25 wt% TAP	0.05	0.01
Petroleum Base Stock	AL-10134	0.075	0.09
	AL-10134 + 0.1 wt% Zn	0.032	0.295
	AL-10134 + 0.2 wt% Zn	0.04	0.05
	AL-10134 + 5 wt% TAP	0.057	0.067
	AL-10134 + 15 wt% TAP	0.052	0.06
	AL-10134 + 25 wt% TAP	0.06	0.08
Oil H TEO	AL-14558-L	0.05	0.4
	AL-14558-L + 0.1 wt% Zn	0.06	0.4
	AL-14558-L + 0.2 wt% Zn	0.06	0.4
	AL-14558-L + 5 wt% TAP	0.05	0.1
	AL-14558-L + 15 wt% TAP	0.03	0.065
	AL-14558-L + 25 wt% TAP	0.09	0.4
Oil J TEO	AL-14601-L	0.05	0.35
	AL-14601-L + 0.1 wt% Zn	0.057	0.12
	AL-14601-L + 0.2 wt% Zn	0.065	0.067
	AL-14601-L + 5 wt% TAP	0.06	0.425
	AL-14601-L + 15 wt% TAP	0.07	0.37
	AL-14601-L + 25 wt% TAP	0.075	0.415

* AL-6185-L.

** AL-12072-L.

PAO base stock performance considerably; however, a small amount of TCP has an adverse effect on ester-based oils. Both of the tested additives have deleterious effects in all concentrations on the ester-based oil. Oil H, one of the two turbine oils tested, did not show any improvement due to zinc while showing an improved performance due to TCP. The effect of the two additives on the Oil J turbine oil was, however, the reverse, i.e., the 5 wt% TCP deteriorated the performance of the neat oil but steadily improved friction coefficient with increasing zinc. In most cases, there was an optimum additive concentration that was dependent on base stock type.

A Taylor-Hobson Talysurf-10 instrument was set up to measure wear tracks produced by the Cameron-Plint rig. Initial data from the Talysurf instrument revealed that wear tracks from the hard tool steel specimens were below detection limits for the experimental conditions reported in TABLE 31. Additional work is planned for the Cameron-Plint rig using specimens cut from actual diesel engine ring and liner segments, which will give measurable wear in a reasonable test time.

V. HIGH-TEMPERATURE LUBRICANT ANALYSIS TECHNIQUES

A. Methodologies

A methodology for analyzing a new high-temperature lubricant was developed. A flow chart that illustrates the new lubricant analysis techniques is shown in Fig. 18. The flow chart shows that a new oil is analyzed for chemical/physical properties by standard American Society for Testing and Materials (ASTM) tests and various instrumental analysis techniques. Performance tests for oxidation, deposition, and friction/wear are also used to characterize the lubricant. Also shown are the generalized procedures for additive and base stock analysis. In Fig. 18, the detailed ester base stock analysis techniques are shown, which were previously detailed in Reference 21. A flow chart for detailed used oil analysis is presented in Fig. 19. Performance bench tests are utilized in the analysis of used oils to determine the degradation in performance after service and to give an indication of the remaining lubricant life.

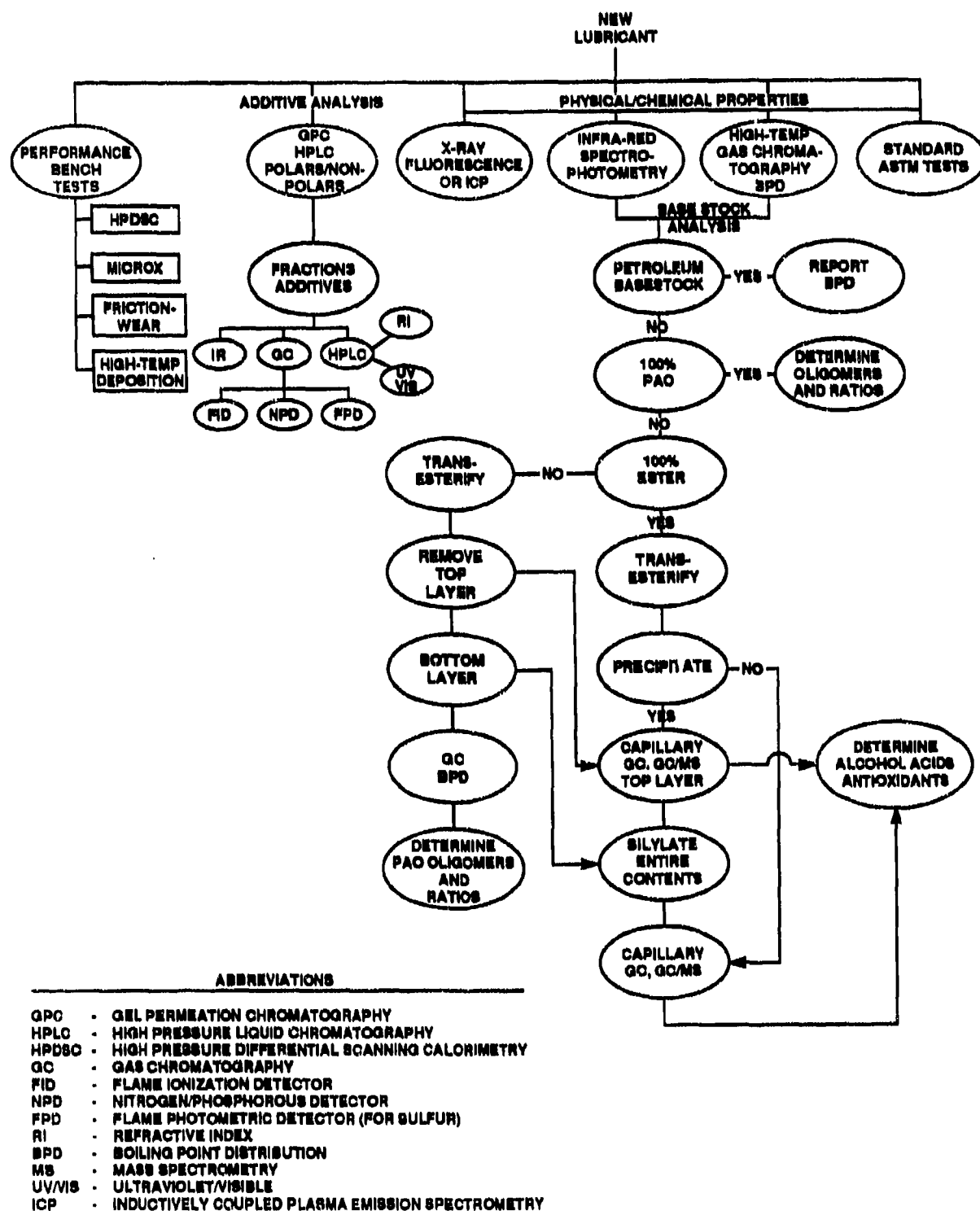


Figure 18. New lubricant analysis techniques

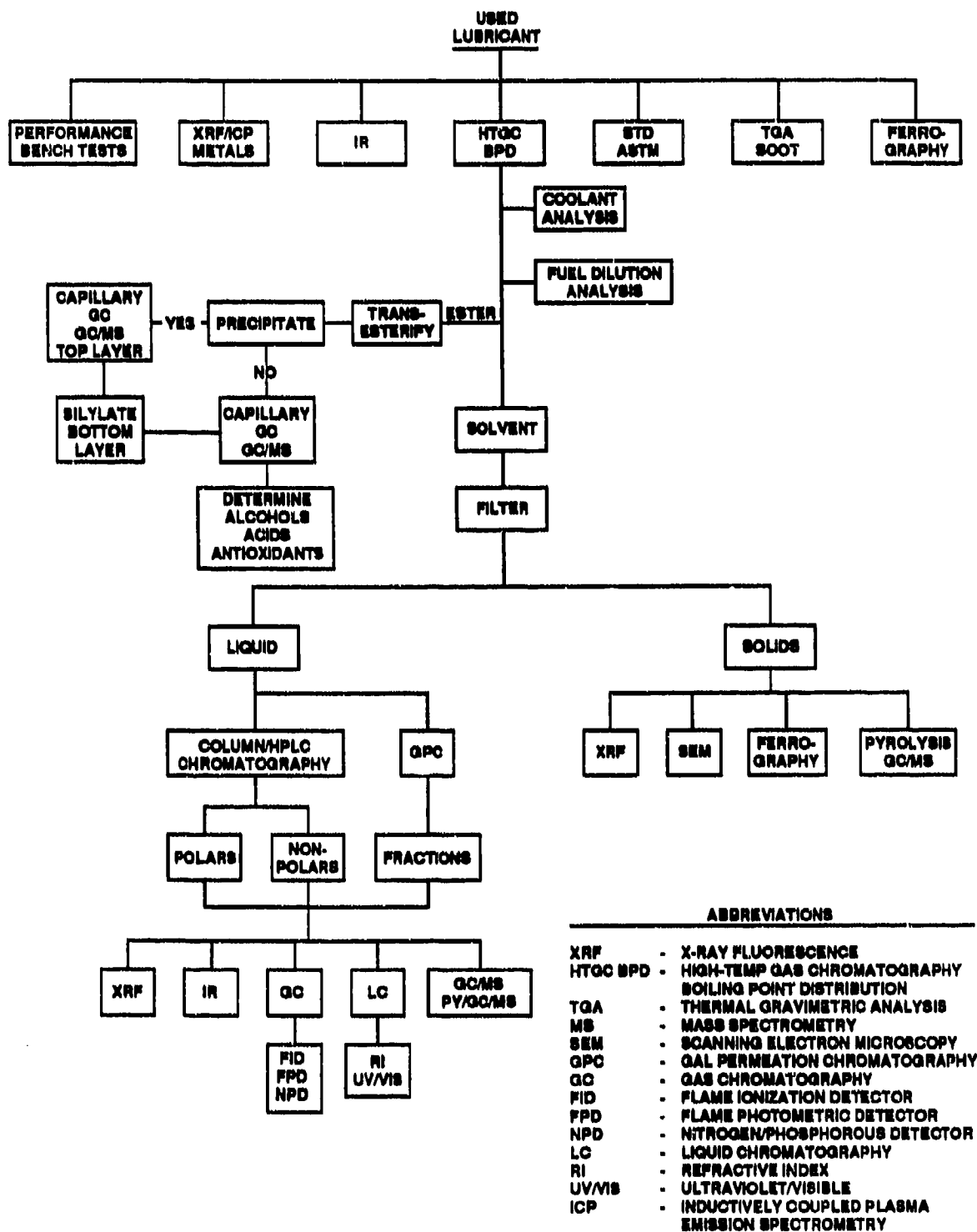


Figure 19. Used lubricant analysis techniques

Additional detailed analysis of lubricant additive compositions were developed for viscosity index improvers and zinc dithiophosphate additives. Zinc dithiophosphate additives have been separated from base oil using High Pressure Liquid Chromatography (HPLC) with a silica gel column to collect polar and nonpolar fractions. The polar fraction, containing the zinc additives, is analyzed by infrared to distinguish between primary alkyl, secondary alkyl, and aryl ZDP additives.

A Gel Permeation Chromatographic (GPC) separation of a lubricant followed by infrared analysis of the high molecular weight fraction gives approximate molecular weight and type identity of the VI improver. Polymethylacrylate, isoprene/styrene, and olefin copolymer Type VI improvers have been investigated to date (TABLE 32). They were analyzed by infrared spectrophotometry for functional group and chemical type and wide-range gel permeation/size exclusion (GPC/SEC) liquid chromatography to obtain an approximate molecular weight/molecular size at ambient temperature. The approximate molecular weight (MW) of the separated Viscosity Index Improvers (VII) from each additive sample was calculated following analysis on a mixed bed GPC column. The approximate MW was calculated versus polymethylmethacrylate standards and polystyrene standards (TABLE 32). Sample AL-14467-A was dropped from further analysis because the molecular weight indicated it was primarily used as a pour point depressant rather than a VII. More precise MW determinations could be made using a narrower range of GPC columns. The infrared (IR) spectrum of each fraction was recorded. Blends of each additive at 5 to 8 wt% in a petroleum-base oil were compounded and analyzed by IR. Their spectra were

TABLE 32. Analysis of the Viscosity Index Improvers

BFLRF No.	Type	Calculated MW	
		Using Polymethyl Methacrylate Std	Using Poly-Styrene Std
AL-13364-A	Nondispersant Polymethylacrylate	363,000	453,000
AL-14467-A*	Polymethacrylate	63,000	57,000
AL-14468-A	Dispersant Polymethylacrylate	323,000	428,000
AL-14469-A	Isoprene/Styrene	273,000	375,000
AL-14470-A	Nondispersant Olefin Co-Polymer	242,000	325,000

* Primary use is as a pour point depressant - No further analysis planned.

studied to determine if the VII type can be determined directly in a lubricant. Preliminary indications are that the methacrylate and isoprene/styrene types can be determined in the whole oil. The olefin type will require additional techniques. GPC analysis of the base oil/VII blends indicated no difficulty in separating the high MW VII.

B. Oil Characterizations

Five of the high-temperature lubricants (Oils D, E, F, G, and H) were characterized to determine their base stock composition using the analytical scheme developed by BFLRF.(20) The chemical characterization of the base stocks of Oils D, E, and F is summarized in TABLE 33. Included are the oligomer compositions of five representative PAO base stocks. The residue

TABLE 33. Lubricant Characterization

Base Stock	Composition		Bottom Layer	PAO Composition				Residue, Wt%
	Wt% PAO & Other Materials	Wt% Diester		Ratio Wt%				
				C ₂₀	C ₃₀	C ₄₀	C ₅₀	
Oil E	75	25*	Oil E	3	20	39	9	22
Oil D	50	50*	Oil D	<1	23	19	3	54
Oil F	40	60*	Oil F	<1	7	30	13	49

Top Layer	Ester Composition			Source X	Composition				
	Acids	Vol%	Alcohols						
Oil E	Di-C ₅	65	Decyl	PAO 2CS	99.5	0.5	--	--	0.0
	Di-C ₉	35		PAO 4CS	3	75	--	--	3
Oil D	Di-C ₅	50	Decyl	PAO 6CS	<1	29	33	9	29
	Di-C ₉	50							
Oil F	Di-C ₅	60	Tri- Decyl	Source Y	Composition				
	Di-C ₉	40		PAO 4CS	--	85	11	--	4
				PAO 6CS	--	32	44	5	19

* By difference.

amount indicates high molecular weight material, possibly high molecular weight PAO and/or additive package materials, not eluted in the gas chromatographic analysis. XRF analysis of the layers formed in the chemical characterization shows that the metal contained in the additives remain in the bottom (PAO) layer of these lubricants. All three oils were blends of diester and PAO base stock.

Results of the base stock characterization of the two ester-type turbine lubricants (Oils H and G) that were evaluated in the VM diesel engine are presented in TABLE 34. Oil H was based on trimethylolpropane esterified with primarily iso-pentanoic, n-pentanoic, and nonanoic acids. Oil G contained mostly pentaerythritol and some dipentaerythritol esterified primarily with iso- and normal pentanoic, heptanoic, and nonanoic acids. In summary, improved oil analysis methodology (flow charts), and chemical characterization of base stocks, and some additives were developed. Additional method development is needed to fully identify additive package components.

TABLE 34. Base Stock Characterization of Polyol Ester Components

	<u>Oil H</u>	<u>Oil G</u>
<u>Mono-Carboxylic Acids - wt%</u>		
i-C ₅	22.6	10.6
n-C ₅	39.2	31.6
n-C ₆	1.6	1.7
n-C ₇	2.2	39.4
n-C ₈	3.1	--
n-C ₉	29.5	15.8
n-C ₁₀	0.7	0.5
n-C ₁₁	0.7	0.2
n-C ₁₂	0.3	0.2
<u>Polyols - wt%</u>		
TMP	100	--
PE	--	86.6
DPE	--	13.4

VI. CONCLUSIONS

The following conclusions are offered based on this work:

- The modified VM diesel engine was a useful tool to simulate the operation of low-heat rejection (LHR) engines and to develop lubrication requirements for LHR diesel engines.
- The HT VM engine discriminated HTL deposition performance.
- Fourteen different high-temperature lubricant candidates were evaluated at a variety of cylinder wall temperatures ranging from 470° to 650°F (243° to 343°C).
- None of the HTLs evaluated at 650°F (343°C) CWT had satisfactory performance.
- At 630°F (332°C) CWT, Oil G had satisfactory performance for 77 hours and proved to be the best overall HTL candidate. Oils B and A had the next best overall performance and were marginally acceptable at 600°F (316°C) CWT.
- Ring zone oil collection and analyses revealed that oil degradation in the ring zone was accelerated. At 550°F (288°C) CWT, oil degradation was as much as 3.7 times more severe than sump oil degradation. At CWTs greater than 600°F (316°C), the following types of oil degradation problems were observed for some of the oils: excessive viscosity increase, TBN and additive metal accumulations, viscosity decrease from molecular cracking at high-temperature and excessive TAN accumulation.
- A modified Method 5307 glassware oxidation-corrosion was useful in screening HTL candidates, but did not always order oils the same as their oxidation performance in the HT VM engine.

- Initial work using the Cameron-Plint high-frequency friction-wear rig indicated that the effect of ZDTP and TCP additives on friction coefficient was dependent on the type of base stock involved.
- New and used oil analysis flow charts were developed. These flow charts provide guidelines for in-depth analysis on both new and used oils.
- Techniques to separate and identify HTL additives and base stocks were developed.

VII. RECOMMENDATIONS

Based on the results of this work, the following recommendations for follow-on effort are offered:

- HTLs with improved high-temperature oxidation and deposition resistance need to be developed.
- Improved additive system chemistries are needed to handle the high CWTs of LHR engines.
- Improved single-cylinder HTL test engine is needed, which is a prototype of a production LHR engine. The single-cylinder 903 engine will be used for this purpose.
- A methodology for evaluating HTL friction-wear characteristics using the Cameron-Plint rig needs to be developed.
- Improved oil oxidation bench test screening is needed. Pressure differential scanning calorimetry and the thin-film microoxidation test are potential methods.
- A reliable bench-scale oil deposition screening method that correlates with LHR engine deposition is needed.

- Improved HTL additive separation and characterization techniques need to be developed, especially for ashless dispersants.

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LIST OF ABBREVIATIONS AND ACRONYMS

API	- American Petroleum Institute	KOH	- Potassium Hydroxide
ASTM	- American Society for Testing and Materials	LHR	- Low-Heat Rejection
Belvoir RDE Center	- U.S. Army Belvoir Research, Development and Engineering Center	MW	- Molecular Weight
BFLRF	- Belvoir Fuels and Lubricants Research Facility (SwRI)	OD	- Outer Diameter
BSFC	- Brake Specific Fuel Consumption	ox-cor	- Oxidation-Corrosion
CLR-D	- Coordinating Lubricant Research-Diesel	PAO	- Polyalphaolefin
CWT	- Cylinder Wall Temperature	RZO	- Ring Zone Oil
DIR	- Differential Infrared	SAE	- Society of Automotive Engineers
EOT	- End-of-Test	TAN	- Total Acid Number
FTM	- Federal Test Method	TBN	- Total Base Number
FTMS	- Federal Test Method Standard	TCP	- Tricresylphosphate
GCBPD	- Gas Chromatography Boiling Point Distribution	TEO	- Turbine Engine Oil
GPC	- Gel Permeation Chromatographic	TRR	- Top Ring Reversal
GPC/SEC	- Gel Permeation/Size Exclusion	VI	- Viscosity Index
HPLC	- High Pressure Liquid Chromatography	VII	- Viscosity Index Improvers
HTL	- High-Temperature Lubricants	VM	- Engine Manufacturer
ICP	- Inductively Coupled Plasma Emission Spectrometry	WTD	- Weighted Total Deposits
IR	- Infrared	XRF	- X-Ray Fluorescence
		ZDTP	- Zinc Dithiophosphate

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